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1296811

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March 16, 2005

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APPLICATION NUMBER: ***60/525,741***
FILING DATE: ***December 01, 2003***
RELATED PCT APPLICATION NUMBER: ***PCT/US04/40074***

Certified by

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PROVISIONAL APPLICATION FOR PATENT COVER SHEET

This is a request for filing a PROVISIONAL APPLICATION FOR PATENT under 37 CFR 1.53(c).

Express Mail Label N .

INVENTOR(S)

Given Name (first and middle [if any])	Family Name or Surname	Residence (City and either State or Foreign Country)
Tompa, Gary S. Rice, Catherine E. Sbrockey, Nick M. Provost, Lloyd G.		Bell Mead, NJ Scotch Plains, NJ Gaithersburg, MD GlenRidge, NJ

☐ *Additional inventors are being named on the _______ separately numbered sheets attached hereto*

17548 U.S. PTO 60/525741 112403

TITLE OF THE INVENTION (500 characters max)

Passive and Active Optical Film Structure(s) Making

Direct all correspondence to: **CORRESPONDENCE ADDRESS**

☐ Customer Number [ ] *Type Customer Number here* → *Place Customer Number Bar Code Label here*

OR

☐ Firm *or* Individual Name	Structured Materials Industries, Inc.				
Address	201 Circle Drive North, Unit 102/103				
Address					
City	Piscataway	State	NJ	ZIP	08854
Country	U.S	Telephone	732-302-9274	Fax	732-302-9275

ENCLOSED APPLICATION PARTS ***(check all that apply)***

☑ Specification *Number of Pages* [ ]
☐ Drawing(s) *Number of Sheets* [ ]
☐ Application Data Sheet. See 37 CFR 1.76
☐ CD(s), Number [ ]
☐ Other (specify) [ ]

METHOD OF PAYMENT OF FILING FEES FOR THIS PROVISIONAL APPLICATION FOR PATENT

☑ Applicant claims small entity status. See 37 CFR 1.27.
☑ A check or money order is enclosed to cover the filing fees
☐ The Commissioner Is hereby authorized to charge filing fees or credit any overpayment to Deposit Account Number: [ ]
☐ Payment by credit card. Form PTO-2038 is attached.

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The invention was made by an agency of the United States Government or under a contract with an agency of the United States Government.
☐ No.
☐ Yes, the name of the U.S. Government agency and the Government contract number are: ____________________

Respectfully submitted,

SIGNATURE [signature] 11/24/03

Date 11/24/2003

TYPED or PRINTED NAME Dr. Gary S. Tompa

TELEPHONE 732-302-9274

REGISTRATION NO. *(if appropriate)* [ ]
Docket Number: [ ]

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PROVISIONAL APPLICATION COVER SHEET
Additional Page

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Docket Number	

INVENTOR(S)/APPLICANT(S)		
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Number 2 of 2

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FEE TRANSMITTAL for FY 2003

Effective 01/01/2003. Patent fees are subject to annual revision.

[✔] Applicant claims small entity status. See 37 CFR 1.27

TOTAL AMOUNT OF PAYMENT ($)

Complete if Known	
Application Number	
Filing Date	
First Named Inventor	
Examiner Name	
Art Unit	
Attorney Docket No.	

METHOD OF PAYMENT *(check all that apply)*

[✔] Check [ ] Credit card [ ] Money Order [ ] Other [ ] None

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FEE CALCULATION

1. BASIC FILING FEE

Large Entity Fee Code	Large Entity Fee ($)	Small Entity Fee Code	Small Entity Fee ($)	Fee Description	Fee Paid
1001	750	2001	375	Utility filing fee	
1002	330	2002	165	Design filing fee	
1003	520	2003	260	Plant filing fee	
1004	750	2004	375	Reissue filing fee	
1005	160	2005	80	Provisional filing fee	80

SUBTOTAL (1) ($) 80

2. EXTRA CLAIM FEES FOR UTILITY AND REISSUE

		Extra Claims		Fee from below		Fee Paid
Total Claims		-20** =		X		=
Independent Claims		- 3** =		X		=
Multiple Dependent						=

Large Entity Fee Code	Large Entity Fee ($)	Small Entity Fee Code	Small Entity Fee ($)	Fee Description
1202	18	2202	9	Claims in excess of 20
1201	84	2201	42	Independent claims in excess of 3
1203	280	2203	140	Multiple dependent claim, if not paid
1204	84	2204	42	** Reissue independent claims over original patent
1205	18	2205	9	** Reissue claims in excess of 20 and over original patent

SUBTOTAL (2) ($) 0

***or number previously paid, if greater; For Reissues, see above*

FEE CALCULATION (continued)

3. ADDITIONAL FEES

Large Entity Fee Code	Large Entity Fee ($)	Small Entity Fee Code	Small Entity Fee ($)	Fee Description	Fee Paid
1051	130	2051	65	Surcharge - late filing fee or oath	
1052	50	2052	25	Surcharge - late provisional filing fee or cover sheet	
1053	130	1053	130	Non-English specification	
1812	2,520	1812	2,520	For filing a request for *ex parte* reexamination	
1804	920*	1804	920*	Requesting publication of SIR prior to Examiner action	
1805	1,840*	1805	1,840*	Requesting publication of SIR after Examiner action	
1251	110	2251	55	Extension for reply within first month	
1252	410	2252	205	Extension for reply within second month	
1253	930	2253	465	Extension for reply within third month	
1254	1,450	2254	725	Extension for reply within fourth month	
1255	1,970	2255	985	Extension for reply within fifth month	
1401	320	2401	160	Notice of Appeal	
1402	320	2402	160	Filing a brief in support of an appeal	
1403	280	2403	140	Request for oral hearing	
1451	1,510	1451	1,510	Petition to institute a public use proceeding	
1452	110	2452	55	Petition to revive - unavoidable	
1453	1,300	2453	650	Petition to revive - unintentional	
1501	1,300	2501	650	Utility issue fee (or reissue)	
1502	470	2502	235	Design issue fee	
1503	630	2503	315	Plant issue fee	
1460	130	1460	130	Petitions to the Commissioner	
1807	50	1807	50	Processing fee under 37 CFR 1.17(q)	
1806	180	1806	180	Submission of Information Disclosure Stmt	
8021	40	8021	40	Recording each patent assignment per property (times number of properties)	
1809	750	2809	375	Filing a submission after final rejection (37 CFR 1.129(a))	
1810	750	2810	375	For each additional invention to be examined (37 CFR 1.129(b))	
1801	750	2801	375	Request for Continued Examination (RCE)	
1802	900	1802	900	Request for expedited examination of a design application	

Other fee (specify) ____________

*Reduced by Basic Filing Fee Paid

SUBTOTAL (3) ($) 0

SUBMITTED BY				(Complete *(if applicable)*)
Name (Print/Type)	Dr. Gary S. Tompa	*Registration No. (Attorney/Agent)*		*Telephone* 732-302-9274
Signature	[signature]			*Date* 11/24/03

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STRUCTURED MATERIALS INDUSTRIES, INCORPORATED
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732 302 V9274 F9275

BUILDING A BETTER WORLD THROUGH SCIENCE, UNDERSTANDING AND COMMERCIALIZATION

November 24, 2003

Box Provisional Application
Assistant Commissioner for Patents
Washington, D.C. 20231

RE: Patent provisional

Dear Patent Officer,

Enclosed Please find our provisional patent application including:

1. Provisional Patent Applications SBIR proposals numbered 41413, 41415, 41469, 41474
2. All incremental and final reports for 41413 and 41415
3. Some supplemental viewgraphs
4. A set of draft claims
5. Other support documents
6. Payment

Sincerely,

Dr. Gary S. Tompa

Passive and Active Optical Film Structure Making

Tompa, Rice, Sbrockey, Provost

11/23/03 & all 4 proposals → 41413, 41415, 41469, 41474 & misc, figure system schematics.

Claims for $LiNbO_3$ MOCVD process

A system for depositing $LiNbO_3$ films, both amorphous and crystalline

~~and process~~

A system for controlling composition to give stoichiometric or congruent $LiNbO_3$ or compositions in between as desired.

– a system for controlling composition to a defined function

A system for controlling uniformity.
– temperature
– precursor flux

A process for depositing amorphous $LiNbO_3$.

A process for depositing crystalline $LiNbO_3$.

A process for depositing Li-containing complex oxides other than $LiNbO_3$.

A process for depositing thick (>1 micron) Li-containing oxides.

A process for depositing oriented crystalline $LiNbO_3$.

These processes wherein selected precursors are dissolved in a solvent that is brought into a flash evaporator (or heated element) to convert it to vapor that is transferred to ~~the~~ a reactor containing a heated substrate whereat a thin film grows (see schematic figures). One or several sources and variable rates with conventional or gas sources may be used to vary ~~or~~ ~~vary~~ the film structure

A process for depositing $LiNbO_3$ doped-as-grown with titanium or other metals to tailor the index or other properties.

A process for depositing $LiNbO_3$ with very low Fe impurities for resistance to optical damage due to the photorefractive effect.

A process for depositing an epitaxial crystalline film on crystalline substrates such as $LiNbO_3$, sapphire, and others with closest packed oxide layers that can serve as a template. Other potential epitaxial substrates include perovskites such as $LaAlO_3$ and $SrTiO_3$, ZnO, and many others.

A process for depositing $LiNbO_3$ as an amorphous layer on substrates such as $LiNbO_3$ and all others considered for epitaxy, as well as quartz, other silicates, ITO, silicon, basically any solid substrate that does not give rise to excessive interfacial reactions with the film.

A process for depositing $LiNbO_3$ films on metal substrates such as Pt or Ni or Cr.

A process for creating multilayer structures incorporating amorphous or crystalline $LiNbO_3$ layers along with dielectric layers (such as CeO_2 other oxides or non-oxides), conducting layers, or other layers crystalline or amorphous, with the $LiNbO_3$ layer being either underneath or on top of the other layers.

A method for depositing layers with controlled index profile through control of doping profiles. and/or composition.

dielectric overcoats / ~~or~~ layered structures – multi-layered dielectrics

TCO overcoats / ~~or~~ layered structures

Note - Sequence of growing layers may be interspaced with annealing amorphous grown films to single crystals or patterning and etching amorphous films before (or after) annealing to a single crystal or poly

This refers to McCarthy's prior process patent work.

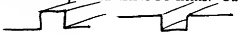
A method for depositing $LiNbO_3$ layers as cladding over existing optical device structures in $LiNbO_3$ bulk or in other optical materials.

<u>Claims for MOCVD $LiNbO_3$ thin film devices</u>

as well as photonic crystal

Active or Passive

Waveguide devices made in amorphous, polycrystalline or epitaxial LiNbO3 films. Can be ridge waveguides or channel waveguides.



Mach-Zehnder type interferometers made in amorphous, polycrystalline or epitaxial LiNbO3 films

Photonic crystal devices made in epitaxial LiNbO3 films,

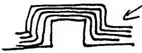
a. using air as the confining medium
b. using doped LiNbO3 as the confining medium
c. using an entirely different material as the confining medium

doped or other material entirely

c.+ multiple layers or combinations of multiple layers

The photonic devices made in LiNbO3 films that are used for:

d. waveguides
e. filters
f. detector
g. mixers
h. other photonic crystal



dielectric stack to reflect light into the waveguide

Light sources made by controlled doping of epitaxial LiNbO3 films with elements such as Er

Optical detectors made by controlled doping of epitaxial LiNbO3 films with elements such as Er

Opto-electronic integrated circuits (OEIC) using LiNbO3 films, either amorphous, polycrystalline or epitaxial, as optical interconnects

Any optical or electro-optical device (functional) built by using a LiNbO3 thin film, with a change in composition, either abrupt or gradual, in which the composition change is used to produce a change in index of refraction, in order to confine a light wave.

Any electro-optical device built by using the property of a LiNbO3 thin film to change its optical properties under the influence of an applied electric field, in order to manipulate an optical signal.

Any electro-optical devices that use a LiNbO3 thin film, as a material with electrically controllable optical properties, in combination with a transparent conductive oxide layer, which is used to apply an electrical field to the LiNbO3. The LiNbO3 film and the transparent conductive oxide may or may not be deposited consecutively in the same deposition chamber.

Any electro . . . copy a dielectric film or stack of films to focus light into the electro-optic device or the selected absence of such films to ~~[illegible]~~ allow light out.

Background Info for Patent

Problem to be solved

- there is a need for devices to control fiber optical communications
  - such as optical switches, modulators
  - also devices for wavelength division multiplexing (WDM)
    - control multiple signals over same fiber
    - multiplexer, with add/drop capabilities

Prior art

- LiNbO3 is best material for electro-optical devices in many cases or near best in other cases
  - high transparency
  - high EO coefficient
  - non-linear optical properties
- There is presently no good process for making thin films of LNbO3
  - presently only bulk devices with diffused layers to define waveguides
    - result is poor confinement
    - large, slow, high voltage devices
- There is a need for a viable thin film technology for LiNbO3
  - Need for both the deposition tools and the process

- Previous attempts to make LiNbO3 thin films
  - films were too thin for practical devices
  - films crack and peeled from substrate
  - deposition rate was too slow.

SMI's approach

- develop hardware and process that can make practical LiNbO3 films
- SMI Hardware solution
  - MOCVD system designed specifically for depositing LiNbO3 thin films
    - Flash Evaporator
    - Rotating Disk Reactor (although any reactor style should work to some level)
    - Close spaced reactor
- SMI reactor can do
  - LiNbO3 deposition rate high enough to be commercially viable
  - thickness is sufficient for waveguide devices,
    - greater than communications wavelength of 1.55 um
  - can do other compositions, including additions such as K, Ta, V, Ti, Fe and any element having a precursor or is evaporable (ie vapor pressure $\gtrsim$ 0.1 Torr at $\lesssim$ 350°C)
  - can do doped films, to raise or lower n
  - can deposit films either amorphous or crystalline
  - can deposit other alloys
  - can be used to anneal the films in-situ

require multiple sources or cocktail solutions

It is preferred the chemical be compatible with the liquid or vapor state of mixing of sources when acceptable and designed structures - some structures may

Run conditions

Precursor and cocktail

Precursors must contain the metals of interest acceptable levels of (lithium, niobium, and any dopants such as (or other alloys) titanium), be sufficiently volatile (vapor pressure at ~~least 1~~ ~0.1 Torr at ~~200~~ ~350°C degrees C), be sufficiently stable (not thermally decompose at the temperature of volatilization), be capable of decomposing to the desired oxide with ~~no~~ unwanted impurities, either with or without an oxidant gas, and be soluble in a suitable solvent if necessary. Examples include niobium penta-ethoxide, lithium tert-butoxide, and titanium iso-propoxide but many others are possible.

Solvents must dissolve the precursors at a suitable concentration (being capable of dissolving to the total metal concentration of 0.01 M) and not react with them to form involatile species. Solvents must also have sufficient vapor pressure to be fully vaporized in the flash evaporator at the conditions used for the precursors. Examples include toluene, tetrahydrofuran, alcohols, and many others. In general, these solvents must be kept free of water and other potentially reactive species.

The cocktail should typically be made up in the absence of water to avoid prereaction of the precursors. The concentration of precursors in solution may range from 0.01 to 1 M; with values in the range 0.05 to 0.2 being especially favorable. Lower concentrations may lead to excessively slow deposition while high concentrations can in some cases lead to inefficient growth or precipitation. This concentration is a variable to be optimized in each specific set of process conditions.

The feed rate of the cocktail into the flash evaporator can range from ~~0.5 to 10~~ ~0.1 to several hundred cc per minute. In practice, rates from 1.3 to 2.5 cc per minute were found to give good results. Too low a rate can lead to drying of the precursor in the lines and clogging (as well as low growth rates), while too high a rate can lead to pooling of solution in the flash evaporator due to inability to volatilize the solution at a fast enough rate (which can give rise to spitting of unvolatilized material through the lines, uneven back pressure, and other unwanted conditions).

Chamber parameters

The following range of run parameters can be used to deposit films in a typical operation

Run parameters	
Substrate temperature	300 - 900° C
FE temperature	200 - 350° C*
Chamber pressure	1 - 100 Torr
Uniform gas (Ar or N_2)	500 - 3000 sccm
Uniform O_2	500 - 5000 sccm
FE push (inert gas)	50 - 200 sccm
Sample rotation	500 – 1000 rpm

Ranges which have been found to deposit good films are:

Run parameters	
Substrate temperature	400 - 625° C
FE temperature	230° C
Chamber pressure	5 - 20 Torr
Uniform inert gas	500 - 3000 sccm
Uniform O_2	1000 - 5000 sccm
FE push (inert gas)	150 - 200 sccm
Sample rotation	750

Amorphous material was found to be deposited at lower temperatures, up to ~450°C substrate temperature. At temperatures of ~475°C or above, the films deposited as crystalline. The films were found to deposit in oriented or epitaxial habit on substrates such as $LiNbO_3$, sapphire, and $LaNiO_3$ and in random orientation on SiO_2 substrates.

For the gases used during deposition, the ones above are given as examples but others are possible. The inert gas can be Ar, N_2, or other unreactive gas. The oxidant is typically oxygen but can alternatively be H_2O, N_2O, alcohols such as CH_3OH, or others. Good results were obtained with the above conditions but the use of enhancements to the MOCVD deposition process – such as plasma, ultraviolet radiation, or other energetic enhancement, can be anticipated to be useful in some circumstances such as if enhanced deposition rate at low temperatures is desired. In addition, plasma or UV light may be used to clean the substrates prior to deposition.

Amorphous $LiNbO_3$ can be advantageous in that it is easily etchable in solutions such as aqueous HF whereas crystalline $LiNbO_3$ is difficult to etch. Thus the deposition of amorphous $LiNbO_3$ is useful in the creation of patterned devices.

Same as all before, but for K Li Nb Ta O

Electro-optic can include ~~any~~ pyroelectric, ferroelectric and other state

41413

Title: A Scaleable Method for Fabricating Nonlinear Photonic Crystals for Ultrafast All-optical and Electro-optic Functions

Abstract:

The University of Wisconsin has developed an MOCVD process by which micron-scale and nanoscale high contrast (air/$LiNbO_3$) epitaxial film photonic structures can be fabricated. As a consequence of the large index difference, as well as the large second order optical nonlinearity of $LiNbO_3$, very compact all-optical and electro-optic devices based on index-confined and photonic bandgap defect waveguides can be realized. These structures will serve as the basis for a new class of truly compact electro-optic and all-optical devices and circuits. The goal of the first phase of this proposed work is to design and demonstrate a reproducible wafer-scale version of the growth process. The follow-on effort will be dedicated to the design, fabrication, and characterization of several devices, including an ultrafast traveling wave modulator, tunable resonator filter, and an all-optical logic switch. This effort will combine the unique expertise of the group of Dr. Leon McCaughan at University of Wisconsin-Madison, discoverer of the technology, Structured Materials Industries, Inc., with extensive knowledge and background in oxide MOCVD processes and equipment, and Pandanus Optical Technologies, for design and fabrication of advanced optical devices. This proposal is addressed to the expressed Air Force need for advanced nanophotonic devices and technology.

Commercial Applications: Successful completion of this program will enable a breakthrough in device capability and quality for a variety of commercial and military applications, including all-optical logic switches, tunable resonator filters and demultiplexers, and very low voltage traveling wave modulators - all part of a growing >$10B market.

KEYWORDS: Lithium niobate, MOCVD, photonic crystal, photonic bandgap, nanophotonics, optical nanodevices, optical switching, modulators

C. Identification and Significance of the Problem or Opportunity

Photonic crystals, structures in which the refractive index is a periodic function in space, show exciting promise for the realization of a variety of novel and improved optical devices, including single-mode LEDs and thresholdless lasers.[1] $LiNbO_3$, with its large index difference and large second order optical nonlinearity, has the potential to significantly extend the functionality of photonic crystal structures to the all-optical and the ultrafast electro-optical regimes – at the sub-mm scale. Device functions include all-optical logic switches, tunable resonator filters and demultiplexers, and very low voltage traveling wave modulators A major factor inhibiting these applications, however, has been a lack of ability to process and fabricate important optical materials into the complex structures required.

$LiNbO_3$ has posed an especially difficult materials processing problem. $LiNbO_3$ is the nonlinear material of choice for performing all-optical and electro-optic functions. Unfortunately the high chemical stability of crystalline $LiNbO_3$ effectively precludes the use of standard photolithographic patterning techniques: wet etching (HF/HNO_3) is limited to several nm/min; dry etching (RIE or RIBE) to 10's of nm/min. The tool set available for processing bulk $LiNbO_3$ is limited to rudimentary functions (e.g., thermal diffusion) on bulk materials. As a consequence, it has not been possible to take advantage of the large refractive index and large nonlinearity of $LiNbO_3$: demonstrated devices are currently based on weakly guiding waveguide-based devices. The successful development of high-index-contrast waveguides and photonic crystal features in nonlinear optical ferroelectrics such as $LiNbO_3$ would serve as the basis for a new class of truly compact electro-optic and all-optical devices and circuits.

Recent results from the McCaughan group at the University of Wisconsin-Madison (UWM) have demonstrated a unique solution to the intractability of crystalline $LiNbO_3$: (1) deposition (via MOCVD) of an amorphous form of $LiNbO_3$, which can be successfully etched, on a crystalline $LiNbO_3$ substrate; (2) etching the amorphous material to form 2-D photonic crystal structures; and (3) annealing the structures to form epitaxial $LiNbO_3$. This work shows tremendous promise of a breakthrough in devices for all-optical signal processing. However, several challenges remain. First, the deposition rate for amorphous $LiNbO_3$ must be increased substantially for practical fabrication of waveguiding layers, and the process must be scaled to full-wafer size to enable commercial scale development.

In this Phase I STTR program, we propose to demonstrate the feasibility of $LiNbO_3$ photonic crystal optical devices on a wafer scale, by combining the strengths of UWM and Structured Materials Industries (SMI). SMI has a long history of developing oxide MOCVD film technology and deposition tools. The Phase I objectives will be to improve deposition rate, taking advantage of recent fundamental mechanistic studies[2] at UWM; modify SMI deposition equipment for the process and transfer the deposition technology to SMI, to show that the process can be carried out in a production scale MOCVD reactor technology; and demonstrate the patternability and optical quality of wafer-scale $LiNbO_3$ epitaxial films. We will also design the commercial $LiNbO_3$ film deposition system. In Phase II, we will build the commercial film deposition system and design, fabricate and characterize several photonic crystal structure devices, including compact, low voltage integrated optical switches and electro-optical modulators. Phase III will consist of system sales and licensed device production.

Successful completion of this program will enable a breakthrough in device capability and quality for a variety of commercial and military applications. As a specific example, the ability to perform intelligent surveillance and target acquisition, to rapidly deploy and process information, and to provide secure command and control communications, is requiring significantly more

rapid and sophisticated data routing and processing than is currently available electronically. Further, large scale long duration satellites are moving to use of all-optical communication networks. The optical devices thus far demonstrated have proven to be impractical for reasons of size, efficiency, speed, and noise. All-optical and electro-optic functions based in LiNbO3 photonic crystal geometries address these deficiencies through reduced size, strong optical confinement, and a large optical nonlinearity. This proposal is addressed to the need for the growth and fabrication of submicron dimensioned photonic heterostructure devices with high dimensional and morphological control in order to enable the monolithic integration of microelectronic and photonic circuits expressed in topic AF02T017, "Nanophotonics".

C.1. Device objectives

Ultrafast all-optical signal processing: Real time optical processing such as packet reading/re-direction for fiber optic systems requires real time logic processes – which in turn requires an optical nonlinearity with no latency. As an elementary example, the AND operation can be formed from the multiplication of two binary (0/1) coded optical signals, $E_j\exp i(\omega_j t)$, j=1,2. The product, taken as either an optical difference- or sum-frequency mixing, is encoded at either the microwave, $E_1E_2\exp i(\omega_1-\omega_2)t$ or optical $E_1E_2 e i(\omega_1+\omega_2)t$ basebands. These two operations can be easily produced by way of one of $LiNbO_3$'s second order nonlinearities. However, at reasonable optical powers, conversion lengths are now ~ several cm because of the large cross section of standard $LiNbO_3$ (diffused) waveguides (~100μm²). Both high-contrast index waveguides and photonic crystal defect waveguides, with their inherently small cross sections (~ 0.2 μm²), can dramatically reduced the required device length: assuming comparable launched power and a recognizing that the nonlinear conversion efficiency goes as $\eta \sim L^2/A_{eff}$, the conversion length is reduced some 20-fold. In addition, a properly designed resonator can be used to further enhance the nonlinear transition over the intrinsic scattering in the cavity (determined by its Q). Fig. 1 is a schematic diagram of an optical AND element, consisting of a pair of intersecting defect waveguides with a defect cavity at their intersection. A typical scattering lifetime is ~ 5ps, corresponding to a $Q \sim 10^3$. Assuming that the energy inside the cavity is ~ 1nJ, the nonlinear decay time is some 300 times faster (more efficient) than the linear scattering process. In the next section, we describe a method for producing 2D photonic crystals in a nonlinear material with the required index contrast (~2:1).

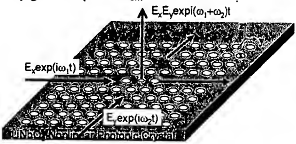


Figure 1. Schematic representation of intersecting defect waveguides in a nonlinear photonic crystal. The d31 coefficient provides the sum frequency product (an optical AND function).

2. *Compact, low voltage integrated optic switches and modulators*: Present-day $LiNbO_3$ integrated optic devices have characteristic lengths ~ several cm, due to the weak confinement of guided light and the consequent large radii of curvature required for device geometries. Using the two-step thin films method we have developed (see below), advantage can be taken of the inherently large refractive index of $LiNbO_3$ to fabricate sub-mm single mode waveguide bends and offsets. The large index contrast, which causes strong optical field confinement, also produces a large microwave field confinement. This combined optical/electrical confinement results in a significant reduction in the operating voltage of electro-optic devices such as traveling wave modulators and optical switch arrays.

We plan to work with device manufacturers such as Pandanus Optical Technologies, Madison WI, and JDS Uniphase (with their worldwide distribution capabilities) to develop both all-optical logic functions as well as low-voltage, ultrafast electro-optic devices such as traveling wave modulators and electro-optically tunable filters.

C.2 Present Technology Status

Two-stage growth method: We have developed a two-stage growth method for fabricating patterned crystalline $LiNbO_3$ structures for photonic crystal and high contrast index waveguide devices, and other photonic applications [patent applied for]. The method uses atmospheric chemical vapor deposition (CVD) to produce an amorphous $LiNbO_3$ film. The film can be patterned using conventional photolithography and standard wet or dry etching techniques, with the crystalline $LiNbO_3$ substrate serving as an etch stop. Alternatively, a standard lift-off process, using SiO_2 as a masking material, can be used to produce a desired pattern. When grown on $LiNbO_3$ substrates, a post-growth anneal converts the amorphous film to single crystal $LiNbO_3$. Figure 3 is a cross-sectional TEM image of an amorphous $LiNbO_3$ film after annealing for 1 hour at 1100°C. The inset is the corresponding [01$\bar{1}$0] zone axis selected area diffraction pattern taken from the film/substrate interface area in the image, demonstrating the single crystal epitaxial nature of the layer. The inclined lines are bend contours; the horizontal band is a thickness fringe.

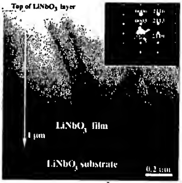


Figure 2. TEM of $LiNbO_3$[3] film after annealing.

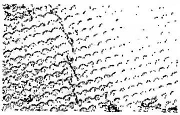


Figure 3. Phase contrast micrograph of an orthorhombic lattice (7.6 x 10.1 μm^2) patterned in $LiNbO_3$

As a preliminary feasibility experiment[3], we grew a thin (~ 2 um) amorphous $LiNbO_3$ film on a z-cut $LiNbO_3$ substrate, patterned it with an orthorhombic 2D periodic pattern (7.6 x 10.1 μm^2), etched in a dilute HF solution, and annealed at 1000°C (Fig. 3). Although not of appropriate dimensions for a linear photonic crystal (~ 0.5 x 0.5 μm^2 needed), the periodicity is appropriate for phase-matched nonlinear functions.

Enhanced deposition rate: A major impediment to the exploitation of $LiNbO_3$ thin films is the consistently low growth rates observed for films grown by means of chemical vapor deposition (CVD) or chemical beam epitaxy (CBE). In the course of our thin film work, we identified what we believe is the major source of the very low deposition rates observed with the commonly-used alkoxides precursors: an autocatalytic cycle involving hydrolysis and dehydration which generates volatile monomers of Li and Nb, instead of stable oxides of the metal.[4,5] Figure 4 shows the cycle for $Li(OBu^t)$. In the absence of this cycle, we estimate growth rates would be some 5-10 times larger than the commonly-observed ~ 0.2 µm/h rate. Film thickness, and therefore growth rate, is a critical issue for photonic devices, since for most applications film dimensions must be greater than, or to the order of, the wavelength of the propagated light ($\lambda \geq 1.5 \mu m$).

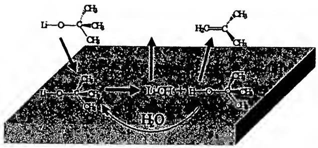


Figure 4. Model for the coupled hydrolysis and dehydration of lithium t-butoxide.

The key to overcoming this defect is to redesign the precursors such that they are either more stable to the presence of water, or decompose by a mechanism that removes the elements of water from the surface before dehydration takes place. In principle, these next-generation precursors can be generated by either of two methodologies: a reactive carrier gas (e.g, Me_3SiCl) reacts to form more reactive precursor intermediates at the growing surface. Alternatively, and more directly, these precursor intermediates can be prepared in the laboratory and then introduced into the growth introduced directly into the growth chamber. Both of these strategies will be tried in this program to find the best alternative for enhancing the $LiNbO_3$ deposition rate.

Finally, it should be noted that we are not limited to growth of congruent ([Li]/[Nb]=0.94) $LiNbO_3$, as in the case of the bulk material. An amorphous form of stoichiometric $LiNbO_3$ should be straightforward to deposit and anneal to crystallinity. Stoichiometric $LiNbO_3$ is known to have significantly smaller susceptibility to the photorefractive effect (i.e., an increase in the material's refractive index with exposure to visible light), and a five-fold lower voltage required for poling (i.e., re-aligning the ferroelectric domains).

C.4 SMI Background MOCVD Technology

At SMI, we have historically used a Low Pressure - Rotating Disk Reactor Metal Organic Chemical Vapor Deposition (LP-RDR-MOCVD) system technology for oxide film deposition. We have recently used this system to produce thin films of Al_2O_3, Cu_2O, SiO_2, MgO, HfO_2, ZnO, InO, ZnO:Ga/In/Al/B/F (transparent-conductive), SrBiTaO, PbZrTiO, CeO_2, $CeMnO_3$, $BaTiO_2$, $SrTiO_2$, and $Ba_xSr_yTiO_2$ (dielectric and pyroelectric); ZnO:Zn, Zn_2GeO_4, and Zn_2SiO_4:Mn. The system technology has previously been used to produce YBCO, YZO, and an array of other oxides, metals, semiconductors, and so on. Uniform films (±5% thickness uniformity or better) over wafers up to 8" in diameter have been achieved, and a system for coating 12" wafers is under construction. Our system technology has great flexibility in producing a wide range of materials, from dopant levels to alloy levels. Importantly, in addition to demonstrating results with Zn, Si, Ga, Al, Mn, In, B, Sr, Ti, Ce, Bi, Sr, Ta, Pb, Zr, Y, Ba, Cu, Hf, and Mg; this system is also well capable of transporting reactants for films containing Nb, La, Ca, Eu, Er, and a host of other materials, i.e., any compound that can be evaporated. Figure 5 shows a schematic diagram of a SMI RDR reactor system. SMI's specialty is working with customers and collaborators to design and construct MOCVD systems tailored to specific oxide film applications. It is this technology platform that SMI brings to the STTR program to design a commercial scale deposition system for high-rate $LiNbO_3$ deposition.

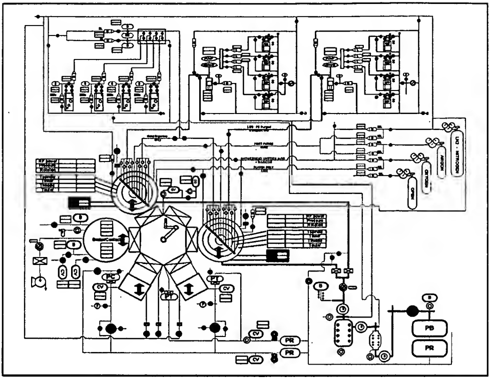


Figure 5. Example configuration for SMI multiple source RDR-MOCVD system. (Note: conventional bubbler, flash evaporators or gas sources may be used in this system.).

C.5. Importance of the STTR Team

The investigators are uniquely suited to carry out this program. A large portion of McCaughan's group at UWM has been devoted to fabrication and characterization of $LiNbO_3$-based nonlinear optics and integrated optics. This group was the first to demonstrate the use of nonlinear photonic crystals for telecommunications applications. Kuech, Saulys, and McCaughan have collaborated extensively in the areas of periodic poling mechanisms and $LiNbO_3$ film growth. Kuech is an internationally known expert both in chemical vapor deposition chemistry and reactor design. The elucidation of the film growth mechanism by Saulys and co-workers have led to the design and synthesis of more effective precursors and reactive carrier gases; these developments have enhanced $LiNbO_3$ thin film growth to the point of enabling growth of prototype device structures. In addition to publications on this collaborative work, the University of Wisconsin has filed provisional patents and plans additional patents.

SMI specializes in process and tool development for commercial scale oxide MOCVD. Dr. Rice is presently leading oxide film growth in ferroelectric oxides such as PZT, PLZT, and CMO, among other oxide MOCVD projects, and Dr. Tompa oversees film growth and directs MOCVD tool development (and has extensive experience with MOCVD of metal oxides and compound semiconductors). No other company has done

as much to advance the state of the art of complex oxide film growth for high technology applications. SMI is thus the ideal partner to work with UWM to produce $LiNbO_3$ films reliably and economically and to develop a tool for commercial production.

Pandanus Optical Technologies of Madison WI will license and commercialize the resulting photonic technologies developed under this effort. JDS Uniphase is also poised to commercialize program results.

D. Phase I Technical Objectives

The overall technical goal of this Phase I program is to demonstrate the feasibility of producing high quality lithium niobate photonic crystal structures at wafer scale, and to design a high throughput deposition tool for commercial scale production. To fulfill this goal we have the following technical objectives:

Objective	*Description*	*Time Period*
1	Transfer UWM deposition technology to SMI	**Months 1 - 2**
2	Optimize amorphous $LiNbO_3$ deposition using SMI scalable test MOCVD reactor.	**3 - 6**
3	Demonstrate processability of films	**7**
4	Refine annealing process for photonic crystal structures	**7 - 8**
5	Perform chemical and optical characterization of films to determine their suitability for guided wave, electro-optic and photonic bandgap devices.	**8 - 10**
6	Design commercial scale MOCVD system dedicated to $LiNbO_3$ process, plan Phase II program	**11 - 12**
7	Write and deliver final report	**12**

E. Phase I Work Plan

E.1 Task 1: Transfer UWM deposition technology to SMI

Drs. Kuech, McCaughan, and Saulys of the University of Wisconsin will work with SMI engineers to design the delivery system for the alkoxides and ethoxide precursors. The SMI scalable research MOCVD reactor will be modified for the specific temperature, pressure, and chemical handling requirements of the UWM process. In particular, some system parts will need to be retooled to be compatible with the chlorine-containing precursors or enhancement agents to be used. UWM and SMI personnel will work together to adapt the deposition process to the rotating disk reactor (RDR) configuration. The UWM process will be demonstrated in the SMI MOCVD system.

E.2 Task 2: Optimize $LiNbO_3$ Deposition

The objective of this task is to enhance the $LiNbO_3$ deposition rate by incorporating improvements delineated by UWM researchers in their fundamental mechanistic study of the deposition process. This study demonstrated that desorption of Li and Nb species was

autocatalyzed by water released by the decomposition of the alkoxide precursors used. Two potential solutions to this problem were identified: addition of a small quantity of trimethylsilylchloride (Me_3SiCl) during deposition using standard alkoxides, or utilizing new precursors designed at UWM. These will be synthesized at UWM and tested as precursors in their own right. The goal is to grow amorphous $LiNbO_3$ at minimum rate of 5 μm/h. Both congruent and stoichiometric thin film compositions will be examined for relative compositional stability. A range of pressure, temperature and flow conditions will be explored to determine the optimum deposition conditions.

E.3 Task 3: Demonstrate processability

In this task, samples of amorphous $LiNbO_3$ films prepared in Task 2 will be tested for etching rates and quality of etched structures. It will be important to determine if the material prepared under enhanced deposition conditions will behave as expected based on previous experience, and to develop procedures for fabricating device structures.

E.4 Task 4: Optimize annealing process

The Phase I efforts will continue to refine the annealing process initiated by UWM in order to define manufacturing needs. Because $LiNbO_3$ is subject to Li_2O outdiffusion during high temperature anneals, it may be necessary to design an annealing station which contains an overpressure of Li_2O during the 1100° C annealing process for Phase II. This may be possible to accomplish by annealing the samples in an atmosphere created by an excess of $LiNbO_3$ powder; this is the simplest technique, and ensures that excessive partial pressures of Li_2O are not present. If this is not sufficient to prevent outdiffusion, a more complex annealing station will be designed and built.

E.5 Task 5: Chemical and optical characterization

Chemical characterization will be performed using SIMS. Structural characterization will be made principally via X-ray diffraction, with a limited number of TEMs to determine defect density. Maker Fringe analysis (a form of surface second harmonic generation) will be used to determine and monitor the Li/Nb ratio. Optical characterization will consist of fabricating channel waveguides and making propagation loss measurements, and determining the magnitude of the nonlinear optic coefficients via the electro-optic effect (e.g., using a standard Mach Zhender waveguide interferometer). We will perform preliminary nonlinear photonic crystal fabrication using electron beam writing capabilities housed within the UW Center for Nanotechnology.

E.6 Task 6: Design MOCVD system and plan Phase II

The objective of this task is to utilize the results of the Phase I study to design a dedicated MOCVD reactor for the deposition of $LiNbO_3$ films and understand processing issues in a manufacturing environment. The Phase II program will also be planned in detail. A tentative outline of tasks anticipated for Phase II is:

1. Build pilot scale reactor system at SMI for $LiNbO_3$ thin film deposition.
2. Optimize epitaxial deposition for 4-inch wafers with characteristics suitable for integrated optic and photonic crystal applications.
3. Optimize annealing of large area wafers.
4. Chemical and optical characterization of uniformity of crystallized thin films.

41415 MDA/ONR

Small Busin ss Technology Transfer (STTR) Program
Proposal Cover Sheet

Propo al Number:	**B023-0099**	**Agency:**	**BMDO**	DUNS:	787147807
Topic Number:	**BMDO02T-00**			CAGE:	OU100
Proposal Title:	MOCVD System for LiNbO3 Thin Film Waveguide Modulators and Optical Switches				

Firm:

Firm Name: Structured Materials Industries

Mail Address: 120 Centennial Ave.

Piscataway, New Jersey 08854-3908

Website Address: www.structuredmaterials.com

Percentage of work: 53 %

Research Institution:

Name: University of Wisconsin - Madison

Mail Address: Dept. of Elect. & Comp. Eng.
1415 Engineering Drive
Madison, Wisconsin 53706

Website Address:

Percentage of work: 47 %

Proposed Cost: 70000 Phase: I Duration: 6

Business Certification: (Check all that apply)

Are you a small business as described in paragraph 2.3?	**YES**
Number of employees including all affiliates (average for preceding 12 months):	**12**
Is the INSTITUTION a research institute as defined in paragraph 2.4? **University**	**YES**
Are you a socially or economically disadvantaged business as defined in paragraph 2.5?	**NO**
Are you a woman-owned small business as described in paragraph 2.6?	**NO**
Has this proposal been submitted to other US government agencies, or DoD components?	**NO**

If yes, list the name(s) of the agency or component and Topic Number in the space below.

Project Manager/Principal Investigator	**Corp rate Official (Business)**	**Institution Official**
Name: Dr. Nick M. Sbrockey	Name: Dr. Gary S. Tompa	Name: Leon McCaughan
Title: Scientist	Title: President	Title: Prof.
Phone: (732) 885-5909	Phone: (732) 885-5909	Phone: (608) 262-0311
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E-Mail: NSbrockey@starpower.net	E-Mail: GSTompa@aol.com	E-Mail: mccaughan@engr.wisc.edu

For any purpose other than to evaluate the proposal, the data referenced below shall not be disclosed outside the Government and shall not be duplicated, used or disclosed in whole or in part, provided that if a contract is awarded to this proposer as a result of or in connection with the submission of this data, the Government shall have the right to duplicate, use or disclose the data to the extent provided in the funding agreement. This restriction does not limit the Government's right to use information contained in the data if it is obtained from another source without restriction. The data subject to this restriction is contained on the pages of the proposal listed on the line below.

Proprietary Information:

Signature of Principal Investigator   Date   Corporate Business Official   Date   Institution Official

Technical Abstract (Limit your abstract to 200 words with no classified or proprietary information)

Electro-optical modulators and switches are needed for increased speed, capacity and flexibility of modern optical communications systems. The designs for these devices exist, as do materials with suitable electro optical properties, such as LiNbO3. However, their potential has not been realized, due to the limitations of diffused structures in bulk LiNbO3 crystals. Recently, our STTR partner at The University of Wisconsin - Madison (UWM) have demonstrated that high quality epitaxial LiNbO3 thin films can be produced by MOCVD. The UWM team has also invented a simple process for defining patterned structures from these films. This technology opens the way for a new class of electro-optical devices, including compact high-speed modulators and optical switches.

Structured Materials Industries, Inc. (SMI) has a long history developing MOCVD systems for complex oxide films. UWM will work with SMI to transition the epitaxial LiNbO3 film technology to commercial viability. We will also partner with a commercial supplier of electro-optical components, to provide technical guidance to the Phase I/II efforts and commercialize the resulting products in Phase III. Together, this team is well positioned to commercialize LiNbO3 thin film waveguide devices. UWM has invented the needed process technology and SMI will develop the necessary commercial hardware.

Anticipated Benefits/Potential Commercial Applications of the Research or Development. (No classified or proprietary information)

five-fold lower voltage required for poling. We should also be able to obtain engineered doping profiles (e.g. with Er^{3+}) potentially leading to a totally new class of electro-optical devices.

C.4 Device Applications

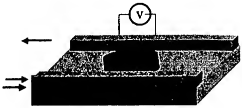

Figure 4: Tunable Ridge Waveguide Ring Resonator

Using the two-step film deposition method, advantage can be taken of the inherently large refractive index of $LiNbO_3$ to fabricate sub-mm single mode waveguide bends and offsets. The large index contrast, which causes strong optical field confinement, also produces a large microwave field confinement. This combined optical/electrical confinement results in a significant reduction in the operating voltage of electro-optic devices, such as traveling wave modulators and optical switch arrays. We plan to work with device manufacturers to develop ultrafast electro-optic devices such as traveling wave modulators and electro-optically tunable filters. The latter is shown schematically in Figure 4. This device is based on high aspect ratio, tightly confining ridge waveguides, which should be easily fabricated using the UWM two stage process. In Phase I, we will demonstrate the feasibility of producing these devices. In Phase II, we will design and fabricate compact, low voltage integrated optic switches and modulators using this technology.

C.5 Importance of the STTR Team

The investigators are uniquely suited to accomplish the goals of this program. McCaughan's group at UWM has been devoted to fabrication and characterization of $LiNbO_3$ based nonlinear optics and integrated optics. This group was the first to demonstrate the use of nonlinear photonic crystals for telecommunications applications. Kuech, Saulys, and McCaughan have collaborated extensively in the areas of periodic poling mechanisms and $LiNbO_3$ film growth. Kuech is an internationally known expert both in CVD processes and reactor design. The elucidation of the film growth mechanism by Saulys and co-workers has led to the design and synthesis of more effective precursors and reactive carrier gases.

SMI is the leading US supplier of MOCVD systems for complex oxide thin films. SMI is currently developing fully integrated systems for Rotating Disc Reactor - Metal-Organic Chemical Vapor Deposition (RDR-MOCVD). We have used this technology to produce thin films and multilayers of a wide variety of complex oxide materials. Of particular interest to this effort is our related work on perovskite materials, such as $BaTiO_3$, $SrTiO_3$, $Ba_xSr_yTiO_2$ $SrBi_2TaO_9$ and $PbZr_xTi_{1-x}O_3$. Refer to Section F.

Pandanus Corporation was founded in 2001 to develop and commercialize the groundbreaking $LiNbO_3$ optical device technologies that originated at the University of Wisconsin. Pandanus original market studies indicate the potential for $500 million in component sales if fundamental $LiNbO_3$ device design limitations could be overcome. Pandanus is also building relationships with companies such as JDS Uniphase, to help with the insertion of these devices into today's fiber-optic telecommunication market. Pandanus Optical Technologies will license and commercialize the resulting photonic products developed under this effort.

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D. Phase I Technical Objectives:

The mission for the UWM / SMI team is to bring the UWM process technology to market. We will accomplish this in two steps. In Phase I, we will demonstrate technical feasibility. In Phase II, we will develop actual products. We will also partner with commercial organizations engaged in the development of fiber optic communications products. Pandanus Optical Technologies, of Madison, WI will provide technical input to the initial stages of this effort, and license and commercialize the resulting electro-optical devices during later program stages.

The UWM / SMI / Pandanus team is uniquely qualified to accomplish the goals of this program. Each brings specific capabilities to the effort. UWM provides the thin film process technology, and materials and device characterizations capabilities. SMI brings expertise in development and commercialization of MOCVD film deposition systems. Pandanus provides knowledge of optical communications technology and products. In addition, we will also partner with JDS Uniphase, who brings worldwide distribution capabilities to this effort.

The following Table summarizes the team's objectives for the Phase I program, as well as for Phase II and Phase III.

Phase I Objectives	
1	Identify critical hardware issues for the UWM MOCVD process and develop specifications for a commercial epitaxial $LiNbO_3$ film deposition system.
2	Demonstrate properties of the resulting epitaxial films that meet the requirements for compact, high-speed electro-optical devices. The Phase I targets are epitaxial $LiNbO_3$ films, at least 2 microns thick, deposited at rates of at least 1 micron/hour, with optical losses of 1 dB/cm or less.
Phase II Objectives	
1	Develop a production worthy MOCVD system for deposition of epitaxial $LiNbO_3$ films, based on the UWM technology. The system will meet all process specifications established in Phase I, as well as ultimate customer expectations for reliability and economical operation.
2	Develop and refine the $LiNbO_3$ MOCVD deposition process, to produce films suitable for electro-optic device fabrication, on wafer sizes up to six inches. The Phase II targets are epitaxial $LiNbO_3$ films, greater than 5 microns thick, deposited at rates of 5 micron/hour or better, with optical losses less than 1 dB/cm.
3	Develop and demonstrate $LiNbO_3$ thin film waveguide devices, including compact high-speed electro-optical modulators and optical switches. Develop strategic partnerships with device manufacturers for licensing and commercialization of the devices.
Phase III Objectives	
1	Commercialize the complete thin film deposition system.
2	Commercialize $LiNbO_3$ based electro-optical modulators and optical switches through strategic partnerships.

E. Phase I Work Plan:

Task Number	*Task Description*	*Month*
1	Transfer the UWM process technology to development MOCVD reactor at SMI.	1 - 3
2	Optimize the MOCVD precursor chemistry for maximum deposition rate and consistent process performance.	2 - 4
3	Refine and demonstrate amorphous $LiNbO_3$ deposition using scalable test MOCVD reactor at SMI.	2 - 4
4	Refine and demonstrate annealing technology to produce epitaxial $LiNbO_3$ films from the amorphous deposits. Characterize the films chemical, physical and optical properties and demonstrate their suitability for waveguide devices.	3 - 5
5	Provide films to electro-optical device manufacturers and develop strategy for device implementation.	4 - 5
6	Design scaled up reactor.	5
7	Deliverable - Phase I final report and Phase II proposal.	6

Task 1: Transfer UWM process technology to development MOCVD reactor at SMI.

UWM will establish a list of critical process issues for MOCVD of epitaxial ferroelectric thin films. This list will define the process requirements in terms of substrate temperature, gas flow rates, precursor chemistry, precursor delivery method, atmospheric moisture tolerance, oxygen activation method and other hardware related issues. This information will be established partly through experimentation and partly from existing process technology at UWM. Drs. Kuech, McCaughan, and Saulys of the University of Wisconsin will work with SMI engineers to facilitate all aspects of the technology transfer, including the design of the delivery system for the alkoxide and ethoxide precursors. The deliverable for this task will be the successful transfer of the UWM process to the development reactor at SMI, and a set of specifications for the production MOCVD system for epitaxial $LiNbO_3$ film deposition.

Task 2: Optimize the MOCVD precursor chemistry for maximum deposition rate and consistent process performance.

UWM will continue with their research to determine the mechanisms and kinetics of $LiNbO_3$ deposition from alkoxides and ethoxide precursors. The proposed UWM process takes advantage of their recent findings regarding the unwanted formation of volatile hydroxides. To avoid this unwanted process, a reactive carrier gas such as Me_3SiCl will be investigated. Preliminary finding by the UWM team show that the introduction of a small amount of Me_3SiCl doubles the growth rate of Nb_2O_5. Experiments will be done to determine if similar results can be obtained for $LiNbO_3$. In addition, UWM has identified the likely precursor intermediates responsible for this increase in growth rate. These intermediates will be synthesized at UWM and evaluated as precursors in their own right. The deliverable for this task will be an assessment of which approach is best for the MOCVD precursor chemistry.

Task 3: Refine and demonstrate amorphous $LiNbO_3$ deposition using scalable test MOCVD reactor at SMI.

SMI will perform process investigations on the development MOCVD reactor, starting with the UWM process and precursors. We will use low cost substrates (such as silicon or sapphire) for the initial process development. We will then use either $LiNbO_3$ or $LiTaO_3$ single crystal substrate, for demonstration of the

optimized epitaxial film properties. The primary intent of this task will be to demonstrate a process to deposit amorphous $LiNbO_3$ films, at least two microns thick, at growth rates of at least one micron/hour. In the course of this activity, we will identify any hardware issues related to the scale-up of the process to commercial wafer sizes and production volumes. We will show proof of concept that we can resolve all scale-up issues. We will also investigate different $LiNbO_3$ film compositions, including both the congruent ([Li]/[Nb] = 0.94) and stoichiometric ([Li]/[Nb] = 1.00) film compositions. Bulk $LiNbO_3$ materials are limited to the congruent composition. However, the MOCVD process should allow us to deposit films of any composition. Selected films from this task will be provided to UWM for evaluation as described in Tasks 4 and 5 below.

Task 4: Refine and demonstrate annealing technology to produce epitaxial $LiNbO_3$ films from the amorphous deposits. Characterize the films chemical, physical and optical properties and demonstrate their suitability for waveguide devices.

UWM will refine and demonstrate the annealing technology to produce epitaxial $LiNbO_3$ films from the amorphous deposits produced at SMI in Task 3. Because $LiNbO_3$ is subject to Li_2O out-diffusion during high temperature anneals, it may be necessary to design an annealing station which contains an overpressure of Li_2O. The goal of this task will be to demonstrate epitaxial films, of 2 micron thickness or greater, without stress related failures such as cracking. We will also demonstrate a process to pattern the amorphous deposits, and anneal them to produce pre-defined structures in the epitaxial $LiNbO_3$ films.

The resulting thin films will be characterized as needed at UWM. Chemical characterization will be made using SIMS. Structural characterization will be made principally via X-ray diffraction, with a limited number of TEMs to determine defect density. Maker Fringe analysis (a form of surface second harmonic generation) will be used to determine and monitor the Li/Nb ratio. These characterization results will be provided as feed-back to the on-going process development work at SMI.

Optical characterization will consist of fabricating channel waveguides and making propagation loss measurements, and determining the magnitude of the nonlinear optic coefficients via the electro-optic effect (e.g. using a standard Mach Zehnder waveguide interferometer). Our targets for this task are to obtain films with optical loss of 1 dB/cm or less, and electro-optic properties approaching that of bulk $LiNbO_3$. UWM is fully equipped to carry out all aspects of this characterization. See Section J for a description of facilities.

Task 5: Provide films to electro-optical device manufacturers and develop strategy for device implementation.

Selected samples of the most promising epitaxial $LiNbO_3$ films will be fabricated into simple waveguide structures and devices at UWM. We will provide samples to our partners at Pandanus Optical Technologies and JDS Uniphase (and other potential customers/partners that we identify) for their evaluation. The deliverable for this task will be verification that the films have the necessary properties for application in electro-optical devices, and a plan for the development of these devices in the Phase II effort.

Task 6: Design scaled up reactor.

Using the results of Tasks 1 through 3, we will establish the design for a commercial MOCVD system, scaling the UWM process technology up to commercial wafer sizes and production volumes. We anticipate that the major design issues will involve the precursor delivery system and the vacuum technology to exclude atmospheric moisture from the deposition chamber. Other engineering considerations in the design include the chamber wall temperature, which must be high enough to prevent precursor condensation but low enough to avoid film deposition. Materials compatibility in the precursor delivery system will also need to be considered. Gas flow effects will need to be evaluated, particularly

buoyancy effects above the disk at the high temperatures. SMI has the engineering expertise, and the computer modeling capabilities (if necessary) to resolve each of these issues. We are well experienced and well qualified in this type of system design. The deliverable for this task will be a set of design specifications for the epitaxial $LiNbO_3$ thin film production system. The actual development of the production system will proceed in Phase II.

Task 7: Reporting.

In accordance with the requirements of the MDA, we will document all of our findings in a final report. If feasibility is demonstrated, we will also present our plans for further development in a Phase II proposal.

Throughout this effort, both SMI and UWM will secure intellectual property rights for the hardware and processes by filing for patents, as appropriate. We will also disseminate technical information through technical publications, conference presentations, trade shows and through our product marketing efforts at SMI.

F. Related Work:

F.1 Related Research at The University of Wisconsin - Madison

Simultaneous all-optical wavelength interchange: Reported the first telecommunication application of 2-D nonlinear lattices: the theoretical demonstration of simultaneous optical wavelength interchange. The two DFG processes essentially "diffract" the interconverted signals from the unconverted ones, providing spatial segregation to eliminate coherent in band cross talk. Presented the first experimental demonstration of simultaneous optical wavelength interchange between the wavelengths 1535 nm and 1555 nm.

Er:Yb:LiNbO3 waveguide optical amplifier: Developed a method for greatly enhancing the local incorporation of Er^{3+} into Ti:$LiNbO_3$ integrated optic devices. In the course of this work identified 4 major sites of Er^{3+} incorporation into $LiNbO_3$. Demonstrated enhanced pump absorption via Yb^{3+} co-doping. Out of this basic research, demonstrated a 980 nm pumped $LiNbO_3$ waveguide optical amplifier.

A rigorous theory for intersecting optical waveguides: Developed a rigorous theory for intersecting optical waveguides which correctly explains and predicts all of the optical characteristics of this waveguide geometry: excellent agreement between calculated and measured coupling constants is found; prediction and observation of a sharply peaked radiation pattern, which in turn explains the anomalously large losses at certain intersection angles; modification of the refractive index of the region common to intersecting waveguides (fractional doping) provides a mechanism for controlling the guided-guided optical coupling characteristics.

Fundamental limits to crosstalk in Ti:$LiNbO_3$ devices: Predicted and subsequently verified that small, randomly distributed, index variations in optical switches produces crosstalk and an asymmetric response.

Three-electrode geometry reduces optical crosstalk in Ti:$LiNbO_3$ switches. We demonstrated with model calculations and by experiment that a 3-segment electrode would provide sufficient degrees of electrical freedom to compensate for the randomly distributed phase mismatch in optical switches and therefore reduce optical crosstalk. We demonstrated a record extinction ratio of -48 dB, almost two orders of magnitude improvement.

Faster integrated optical devices without a power penalty: Our calculations show that the voltage-length product of directional coupler switches is not a constant of the device, as previously presumed (e.g., less

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C. Identification and Significance of the Opportunity:

C.1 Overview

To move beyond the current use of fiber optics as a point-to-point "telegraph" system will require the development of high speed electro-optical modulators and optical switches. $LiNbO_3$ is a nearly ideal material for fabrication of these devices, due to its large refractive index, excellent transparency and electro-optical properties. However, the potential of this material has yet to be realized commercially. Few products have been developed to date, due to the lack of a suitable technology to fabricate $LiNbO_3$ thin film waveguide structures.

Recently, our STTR partners at The University of Wisconsin - Madison (UWM) have developed a two step process for producing waveguide structures in epitaxial $LiNbO_3$ thin films. The patented UWM process results in $LiNbO_3$ films of sufficient thickness and excellent quality, suitable for electro-optical device applications. The UWM process also enables high deposition rates, and relatively simple sub-micron patterning, both of which impact commercial viability in a positive way. The resulting waveguide structures will serve as the basis for a new class of truly compact electro-optic devices, offering greater speeds and lower operating voltages.

In this effort, UWM and SMI propose to work together to transfer this technology to commercialization. UWM will provide the thin film technology. SMI will development the deposition equipment. We will also work with potential end-users, including Pandanus Optical Technologies and JDS Uniphase, for the electro-optical device technology and ultimate device commercialization. Together, this team is well positioned to accomplish the objectives of the proposed effort. In Phase I, we will demonstrate proof of concept for a commercial, wafer-scale version of the UWM process. In Phase II, we will build the commercial film deposition system and design, fabricate and characterize electro-optical devices. In Phase III, we will commercialize the film deposition systems and the resulting electro-optical devices.

C.2 Materials Issues for Electro-optical Devices

Modern communication is increasingly based on fiber optics. This is due to the fact that optical signals carry a higher information content (more bits/second) than conventional electrical signals[1]. To gain even greater capacity, optical networks multiplex many signals, in parallel, over a single fiber, using wavelength division multiplexing (WDM) and dense wavelength division multiplexing (DWDM) technologies. Managing these multiple signals requires compact high-speed, low voltage electro-optical components, such as optical switches and modulators. These components, along with their production scale manufacturing technology, are the focus of this proposal.

Optical waveguides require materials with good transparency at the wavelength of interest, (1.55 microns in current telecommunications systems). Waveguides serve as interconnects in opto-electronic integrated circuits (OEIC's) as well as form the basic components of devices such as optical switches and modulators. Waveguide switches and modulators change optical path length in response to electrical signals. A common example is the Mach-Zehnder interferometer[2]. These components require materials that can change optical properties, in a non-linear fashion, in response to electrical signals. Ferroelectric $LiNbO_3$ is a nearly ideal material for both the OEIC interconnects and electro-optical components, having both excellent transparency and a large electro-optical coefficient.

Present day versions of electro-optical switches and modulators are based on bulk crystals of $LiNbO_3$. A third elemental species (typically titanium) is diffused into the crystal to define waveguide layers in the surface[3]. The concentration profile of these waveguide layers is limited to the typical error-function shaped diffusion profile, and thus only graded index waveguides can be produced. As a consequence of the resulting weak confinement of the guided wave, bulk crystal $LiNbO_3$ devices have characteristic lengths on

the order of several centimeters, and large radii are required for device geometries. The end-result is devices that are large, and consequently slow and require high operating voltages.

Ideally, optical waveguide components would be fabricated from thin films of ferroelectric $LiNbO_3$. A thin film deposition technology, such as metal-organic chemical vapor deposition (MOCVD), would allow fabrication of step index waveguide structures, or any other designed concentration profile. This would provide better confinement of the optical signal, and greater flexibility in the device design. The result would be more-compact devices, with consequent higher speeds, lower operating voltages and greater a degree of device integration.

Previous attempts to make $LiNbO_3$ thin films used sputtering[4,5], laser ablation[6-8], sol-gel processing[9], thermal plasma spray CVD[10,11], liquid phase epitaxy[12,13], chemical beam epitaxy[14] and MOCVD[15-22]. Although there have been many encouraging reports of epitaxial deposition, in general, the films suffer from being too thin and from having excessive optical losses. For effective waveguiding, the films thickness must be on the order of the communication wavelength (presently 1.55 microns). Epitaxial $LiNbO_3$ film deposition on sapphire has only achieved a thickness up to 2000 angstroms, because of cracking caused by the large thermal expansion mismatch with the substrate[17]. $LiTaO_3$ substrates have a much better thermal expansion match with $LiNbO_3$, but have only resulted in films up to 6000 angstroms thick[17]. Effective waveguiding also requires films with very low optical loss. Nominally, losses of less than 1 dB/cm are required. The sources of optical losses in thin films are; scattering by defects in the film, scattering by surface roughness, optical absorption and optical dispersion due to polycrystalline materials[23]. Therefore, low-defect density films are required, with low surface and interfacial roughness. The films must also have high purity and good oxygen stoichiometry (for low optical absorption) and must be single crystalline (for minimum optical dispersion).

Another obstacle to implementing thin film $LiNbO_3$ devices is the lack of an efficient patterning technology. Unfortunately the high chemical stability of crystalline $LiNbO_3$ effectively precludes the use of standard photolithographic patterning techniques. Wet etching (HF/HNO_3) is limited to several nm/min. Dry etching (RIE or RIBE) to 10's of nm/min. In fact, the present tool set available for processing bulk $LiNbO_3$ is limited to rudimentary functions (e.g. thermal diffusion) on bulk materials. As a consequence, it has not been possible to take advantage of the large refractive index and large optical nonlinearity of $LiNbO_3$. Demonstrated devices have thus far been based on weakly guiding waveguide structures. The successful development of a technology for deposition and patterning of high quality $LiNbO_3$ thin films would enable a new class of truly compact electro-optic devices and circuits.

C.3 UWM Two Stage Growth Process

UWM has developed a two-stage growth process for fabricating patterned structures of crystalline $LiNbO_3$ for photonic crystal and electro-optical waveguide devices[24]. The method uses MOCVD to deposit an amorphous $LiNbO_3$ film. The amorphous $LiNbO_3$ layer can be easily patterned using conventional photolithography and standard wet or dry etching techniques. For films deposited on crystalline $LiNbO_3$, the substrate serves as an effective etch stop. For amorphous films grown on $LiNbO_3$ substrates, a post deposition anneal converts the amorphous film to single crystal epitaxial $LiNbO_3$. We assume that similar epitaxial films would be obtained on closely lattice-matched substrates such as $LiTaO_3$.

As a preliminary feasibility experiment, UWM grew a 2 micron thick amorphous $LiNbO_3$ film on a z-cut $LiNbO_3$ substrate. The film was then patterned, with an orthorhombic 2D periodic pattern (7.6 x 10.1 μm^2), using standard photolithography and wet etching in dilute HF. Subsequent annealing at 1000 C resulted in the crystalline epitaxial $LiNbO_3$ structure shown in Figure 1.

Figure 2 is a cross-sectional TEM image of a similar amorphous $LiNbO_3$ film after annealing for 1 hour at 1100 C. The insert is the corresponding [01-10] zone axis selected area diffraction pattern, taken from the

film/substrate interface area in the image, demonstrating the single crystal epitaxial nature of the layer. The inclined lines are bend contours; the horizontal band is a thickness fringe. **To our knowledge, this is the first time a 2 micron thick epitaxial $LiNbO_3$ film has been demonstrated.** The previous largest thickness reported was 0.6 microns for an epitaxial film on $LiTaO_3$[17]. These results demonstrate the capabilities of the UWM two stage process, namely the ability to make epitaxial $LiNbO_3$ films, thick enough for practical waveguide applications, and the ability to readily pattern these films to fine geometries.

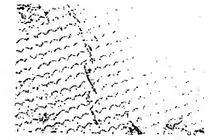

Figure 1: Phase contrast Micrograph of an orthorhombic lattice (7.6 x 10.1 μm^2) patterned in epitaxial $LiNbO_3$.

Another major impediment to the implementation of $LiNbO_3$ thin films is the consistently low growth rates observed for films deposited by MOCVD or chemical beam epitaxy (CBE). Reasonable growth rates are an important consideration for commercial viability. In the course the thin film work at UWM, the researcher identified what they believe is the source of the low deposition rates observed for the commonly used alkoxides precursors[25]. The culprit is an autocatalytic cycle involving hydrolysis and dehydration, which generates volatile monomers of Li and Nb, instead of stable oxides of the metal. Figure 3 shows the cycle for lithium butoxide. In the absence of this cycle, the estimated growth rates for CBE would be some 5 to 10 times larger than the commonly observed; ~ 0.2 um/h rate.

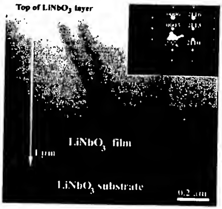

Figure 2: TEM of LiNbO3 film after annealing.

The key to overcoming this defect is to redesign the precursors such that they are either more stable to the presence of water, or decompose by a mechanism that removes the elements of water from the surface before dehydration takes place. In principle, these next-generation precursors can be generated by either of two methodologies. A reactive carrier gas (e.g., Me_3SiCl) can be used to form more reactive precursor intermediates at the growing surface. Alternatively, and more directly, these precursor intermediates can be prepared in the laboratory and then introduced directly into the deposition chamber.

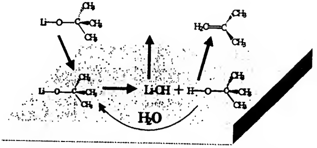

Figure 3: Model for the coupled hydrolysis and dehydration of lithium t-butoxide.

The UWM two stage process results in epitaxial $LiNbO_3$ films, of useful film thickness, and the ability to pattern these films to fine geometries. We also believe we can dramatically increase the deposition rate for $LiNbO_3$, and improve the commercial viability of the process. The other potential advantages of the process are those inherent to MOCVD. We can readily control the composition and composition profile of the films. High purity $LiNbO_3$ films should be achievable. (Bulk $LiNbO_3$ crystals frequently have Fe contamination). In addition, we are not limited to growth of the congruent ([Li]/[Nb]=0.94) composition, as in the case of the bulk material. An amorphous form of stoichiometric ([Li]/[Nb]=1.00) $LiNbO_3$ should be straightforward to deposit and anneal to crystallinity. Stoichiometric $LiNbO_3$ is known to have significantly smaller susceptibility to the photorefractive effect (i.e. an increase in the material's refractive index with exposure to visible light) and a

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For any purpose other than to evaluate the proposal, the data referenced below shall not be disclosed outside the Government and shall not be duplicated, used or disclosed in whole or in part, provided that if a contract is awarded to this proposer as a result of or in connection with the submission of this data, the Government shall have the right to duplicate, use or disclose the data to the extent provided in the funding agreement. This restriction does not limit the Government's right to use information contained in the data if it is obtained from another source without restriction. The data subject to this restriction is contained on the pages of the proposal listed on the line below.

Proprietary Information (list page numbers): 3-25

Signature of Principal Investigator	Date	Corporate Business Official	Date	Institution Official

Technical Abstract (Limit your abstract to 200 words with no classified or proprietary information)

In this Phase I STTR effort, Structured Materials Industries, Inc. (SMI), in collaboration with our academic partners at the University of Wisconsin at Madison (UWM), will develop technology to build chip-scale integrated photonic crystal device networks. The photonic crystal devices will be fabricated in epitaxial lithium niobate ($LiNbO_3$) thin films. This effort we will build on technology invented at UWM, to deposit and pattern epitaxial $LiNbO_3$ thin films.

In this Phase I effort, we will develop the technology to integrate photonic device structures into chip-scale optical networks. We will demonstrate the integration technology by building and testing an integrated electro-optically gated 4-channel add/drop multiplexer.

In Phase II, we will design and fabricate networks containing different optical and electro-optical devices, including lasers, detectors, switches, modulators and multiplexers. The network components will be connected with photonic crystal waveguides, built directly into the epitaxial $LiNbO_3$ thin film. In Phase III, we will commercialize this technology for both government and private sector markets.

Anticipated Benefits/Potential Commercial Applications of the Research or Development. (No classified or proprietary information)

The successful development of this integration technology, combined with our $LiNbO_3$ epitaxial film technology, will enable a direct route to large-scale integrated (LSI) optical device networks. These products will initially find applications in military markets, integrating high-speed communications from command, control and sensor arrays. These products will also meet growing market demands for telecommunications, digital signal processing and all optical computing. These markets are expected to reach multi-billion dollar size by the year 2006.

List a maximum of 8 Key Words or phrases that describe the Project.

$LiNbO_3$
Thin Films
Epitaxy
Patterning
Photonic Crystals
Optical Networks
Add/Drop Multiplexer

Small Business Technology Transfer (STTR) Program
Prop sal C ver Sheet

Proposal Number: F033-0164 Agency: Air Force

DUNS: 787147807
CAGE: OU100

Topic Number: AF03T021

Proposal Title: Chip-scale Photonic Crystal Optical Networks Based on Epitaxial LiNbO3 Thin Films

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Percentage of work: 42 %

Proposed Cost: 100000 Phase: I Duration: 9

Business Certification: (Check all that apply)

Are you a small business as described in paragraph 2.3 (note: wholly owned subsidiaries are not eligible)?	YES
Number of employees including all affiliates (average for preceding 12 months):	12
Is the INSTITUTION a research institute as defined in section 2.4? paragraph 2.4?	YES
University	
Ar you a socially or economically disadvantaged business as defined in paragraph 2.5 ?	NO
Are you a woman-owned small business as described in paragraph 2.6 ?	NO
Are you a certified HUBZone small business concern as described in paragraph 2.12 ?	NO
Has this proposal been submitted to other US government agencies, or DoD components?	NO

If yes, list the name(s) of the agency or component and Topic Number in the space below.

Chip-scale Photonic Crystal Optical Networks Based on Epitaxial $LiNbO_3$ Thin Films

STTR Phase I proposal. Submitted in response to Topic:
AF03T021 "Photonic Crystal Chip-scale Optical Networks"
April, 2003

Part 1: Identification and Significance of the Opportunity:

1.1 Overview

In this Phase I STTR effort, Structured Materials Industries, Inc. (SMI), in collaboration with our academic partners at the University of Wisconsin at Madison (UWM), propose to develop technology to build chip-scale integrated photonic crystal device networks. The photonic crystal devices will be fabricated in epitaxial lithium niobate ($LiNbO_3$) thin films, using standard photolithography and reactive ion etching techniques. $LiNbO_3$ is an excellent material for fabricating photonic crystal devices. $LiNbO_3$ has excellent transparency to the wavelength of interest in optical communications and all-optical computing. In addition, its large second order optical nonlinearity enables highly compact photonic crystal devices. Previously, the full potential of $LiNbO_3$ devices has not been exploited, due to the lack of a suitable deposition and patterning technology for $LiNbO_3$ thin films.

In this proposed effort, we will build on technology invented at UWM to deposit and pattern epitaxial $LiNbO_3$ thin films. During the past year, SMI and UWM have worked together to develop and optimize the UWM process technology and related production hardware. We are also working on technology to build photonic device structures in the resulting epitaxial $LiNbO_3$ thin films. In this effort, we plan to build on these accomplishments. In this Phase I effort, we will develop the technology to integrate photonic device structures into chip-scale optical networks. We will demonstrate the integration technology by building an integrated electro-optically gated 4-channel add/drop multiplexer. This highly compact device will have a footprint of approximately 1 mm^2. The same device made using diffused waveguides in bulk $LiNbO_3$ crystals would have dimensions on the order of 10's of cm^2.

In Phase II, we will further develop and refine the integration technology for $LiNbO_3$ photonic devices. We will design and fabricate networks containing different optical and electro-optical devices, including lasers, detectors, switches, modulators and multiplexers. Many of these components can be constructed directly in the epitaxial $LiNbO_3$ thin film. The network components will be connected with photonic crystal waveguides, also built into the $LiNbO_3$ film. This integration technology, combined with our $LiNbO_3$ epitaxial film technology, will enable a direct route to large-scale integrated (LSI) optical device networks. Also during Phase I and II, we will work with our commercial partners, who include Pandanus Optical Technologies and JDS Uniphase, to plan a commercialization route for the resulting new class of integrated optical devices.

In Phase III, we will design and build integrated optical network devices for military needs. We will also commercialize this technology for both government and private sector markets. We will

commercialize the integrated optical devices through our partners at Pandanus Optical Technologies and JDS Uniphase. Structured Materials Industries will also commercialize the deposition and processing hardware to build these devices.

1.2 Background - Photonic Crystal Devices

Photonic crystals are two and three-dimensional periodic refractive index modulations. The appropriate geometry produces photonic bandgaps, which are frequency ranges where the propagation of light is forbidden inside the structure. Strongly confining channel waveguides can be fabricated by introducing a line of defects in the "crystal." These photonic waveguides can be constructed with very sharp bends and with very low losses[1]. Single-mode LEDs and thresholdless lasers are another consequence of a photonic bandgap lattice's ability to alter the spontaneous emission pattern and rate of an emitting atom[2]. We note that the large refractive indices of ferroelectrics will support 2D air/substrate photonic crystal geometries, giving rise to a new class of low-power, easily integrated, all-optical devices and circuits.

Ferroelectric $LiNbO_3$ is a nearly ideal material for photonic crystal devices, due to its large index difference and large second order optical nonlinearity. Unfortunately, $LiNbO_3$ device development has been limited by the lack of suitable technology for deposition and patterning of $LiNbO_3$ thin films. Present day $LiNbO_3$ devices are fabricated using bulk crystals. A third element (typically titanium) is diffused into the crystal to define waveguide layers in the surface[3]. The concentration profile of these waveguide layers is limited to an error-function shaped diffusion profile, and thus only graded index waveguides can be produced. As a consequence of the resulting weak confinement of the guided wave, bulk crystal $LiNbO_3$ devices have characteristic lengths on the order of several centimeters, and large radii are required for device geometries. The end-result is devices that are large, slow and require high operating voltages.

Ideally, photonic crystal structures could be fabricated in epitaxial thin films of ferroelectric $LiNbO_3$. A thin film deposition technology, such as metal-organic chemical vapor deposition (MOCVD), would allow fabrication of step index waveguide structures, or any other designed concentration profile. With the availability of a suitable pattering technology, photonic bandgap structures could be fabricated in epitaxial $LiNbO_3$ thin film. The resulting photonic crystal devices could perform a wide variety of optical functions, including switching, filtering and modulation. Optical interconnects could also be defined in the $LiNbO_3$ flayer. The resulting integrated devices would be more compact, operate at higher speeds and lower voltages, and provide for a greater a degree of device integration.

Previous attempts to make $LiNbO_3$ thin films have used sputtering[4,5], laser ablation[6-8], sol-gel processing[9], thermal plasma spray CVD[10,11], liquid phase epitaxy[12,13], chemical beam epitaxy[14] and MOCVD[15-22]. Although there have been many encouraging reports of epitaxial deposition, in general, the films suffer from being too thin and from having excessive optical losses. Epitaxial $LiNbO_3$ film deposition on sapphire has only achieved a thickness up to 2000 angstroms, because of cracking caused by the large thermal expansion mismatch with the substrate[17]. $LiTaO_3$ substrates have a much better thermal expansion match with $LiNbO_3$, but have only resulted in films up to 6000 angstroms thick[17]. (As descried in Part 4.1 of this proposal, the UWM/SMI team has recently achieved $LiNbO_3$ films up to 3 microns thick on silicon and z-cut $LiNbO_3$ substrates.)

Effective waveguiding requires films with very low optical loss. Nominally, losses of less than 1 dB/cm are required. The sources of optical losses in thin films are; scattering by defects in the film, scattering by surface roughness, optical absorption and optical dispersion due to polycrystalline materials[23]. Therefore, low-defect density films are required, with low surface and interfacial roughness. The films must also have high purity and good oxygen stoichiometry (for low optical absorption) and must be single crystalline (for minimum optical dispersion). Another obstacle to implementing thin film $LiNbO_3$ devices is the lack of an efficient patterning technology. Unfortunately the high chemical stability of crystalline $LiNbO_3$ effectively precludes the use of standard photolithographic patterning techniques. Wet etching (using HF/HNO_3) is limited to several nm/min. Dry etching (RIE or RIBE) to 10's of nm/min. In fact, the present tool set available for processing bulk $LiNbO_3$ is limited to rudimentary functions (e.g. thermal diffusion) on bulk materials. As a consequence, it has not been possible to take advantage of the large refractive index and large optical nonlinearity of $LiNbO_3$. Demonstrated devices have thus far been based on weakly guiding waveguide structures.

1.3 UWM Two Stage Growth Process

UWM has developed a two-stage growth process for fabricating patterned structures of crystalline $LiNbO_3$ for photonic crystal and electro-optical waveguide devices[24]. The method uses MOCVD to deposit an amorphous $LiNbO_3$ film. The amorphous $LiNbO_3$ layer can be easily patterned using conventional photolithography and standard wet or dry etching techniques. For films deposited on crystalline $LiNbO_3$, the substrate serves as an effective etch stop. For amorphous films grown on $LiNbO_3$ substrates, a post deposition anneal converts the amorphous film to single crystal epitaxial $LiNbO_3$. We assume that similar epitaxial films would be obtained on closely lattice-matched substrates such as $LiTaO_3$.

As a preliminary feasibility experiment, UWM grew a 2 micron thick amorphous $LiNbO_3$ film on a z-cut $LiNbO_3$ substrate. The film was then patterned, with an orthorhombic 2D periodic pattern (7.6 x 10.1 ∝m^2), using standard photolithography and wet etching in dilute HF. Subsequent annealing at 1000 C resulted in the crystalline epitaxial $LiNbO_3$ structure shown in Figure 1.

Figure 2 is a cross-sectional TEM image of a similar amorphous $LiNbO_3$ film after annealing for 1 hour at 1100 C. The insert is the corresponding [01-10] zone axis selected area diffraction pattern, taken from the film/substrate interface area in the image, demonstrating the single crystal epitaxial nature of the layer. The inclined lines are bend contours; the horizontal band is a thickness fringe. **To our knowledge, this is the first time a 2 micron thick epitaxial $LiNbO_3$ film has been demonstrated.** The previous largest thickness reported was 0.6 microns for an epitaxial film on $LiTaO_3$[17]. These results demonstrate the capabilities of the UWM two stage process, namely the ability to make epitaxial $LiNbO_3$ films, thick enough for practical waveguide applications, and the ability to readily pattern these films to fine geometries.

Figure 1: Phase contrast Micrograph of an orthorhombic lattice (7.6 x 10.1 ∝m^2) patterned in epitaxial $LiNbO_3$.

Another major impediment to the implementation of $LiNbO_3$ thin films is the consistently low growth rates observed for films deposited by MOCVD or chemical beam epitaxy (CBE). Reasonable growth rates are an important consideration for commercial viability. In the course

the thin film work at UWM, the researcher identified what they believe is the source of the low deposition rates observed for the commonly used alkoxides precursors[25]. The culprit is an autocatalytic cycle involving hydrolysis and dehydration, which generates volatile monomers of Li and Nb, instead of stable oxides of the metal. Figure 3 shows the cycle for lithium butoxide. In the absence of this cycle, the estimated growth rates for LiNbO3 should be 5 to 10 times larger than the commonly observed; 0.2 μm/hr. Our recent studies of LiNbO3 MOCVD have confirmed these predictions, achieving growth rates up to 3.6 μm/hr. (See Part 4.1 of this proposal).

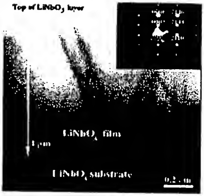

Figure 2: TEM of LiNbO3 film after annealing.

The UWM two stage process results in epitaxial $LiNbO_3$ films, of useful film thickness, and the ability to pattern these films to fine geometries. We have already demonstrated dramatically increases in the deposition rate for $LiNbO_3$, which will improve the commercial viability of the process. The other potential advantages of the process are those inherent to MOCVD. We can readily control the composition and composition profile of the films. High purity $LiNbO_3$ films should be achievable. (Bulk $LiNbO_3$ crystals frequently have Fe contamination). In addition, we are not limited to growth of the congruent ([Li]/[Nb]=0.94) composition, as in the case of the bulk material. An amorphous form of stoichiometric ([Li]/[Nb]=1.00) $LiNbO_3$ should be straightforward to deposit and anneal to crystallinity. Stoichiometric $LiNbO_3$ is known to have significantly smaller susceptibility to the photorefractive effect (i.e. an increase in the material's refractive index with exposure to visible light) and a five-fold lower voltage required for poling. We should also be able to obtain engineered doping profiles (e.g. with Er^{3+}) potentially leading to a totally new class of electro-optical devices.

1.4 Integrated Photonic Crystal Device - Electro-optically Gated Optical Add/Drop Multiplexer

As a first example of integration in nonlinear photonic bandgap (NLPBG) materials, we intend to fabricate a switchable 4-wavelength optical add/drop multiplexer (OADM). The optical circuit will be fabricated using 2D defect waveguides in a photonic crystal (~ 450 nm periodicity). These guides have the advantage of being able to provide very strong optical confinement. As a consequence, abrupt changes in direction of the light and very small radii of curvature, without loss of optical energy, are possible. This will permit a much higher degree of integration than is available with conventional index guides. A schematic of the proposed device is shown in Figure 3.

The degree of confinement is controlled by the number of lattice layers adjacent to the guiding region; evanescent coupling is produced by reducing the number of layers between guides. These waveguides have the added advantage of being able to be fabricated asymmetrically, i.e., with strong optical confinement on one side and weaker confinement on the other (to promote coupling). One of our tasks will be to determine the confinement per lattice layer (~ 1 dB/lattice layer in 3D photonic crystals – S. Fan, personal communication).

We are taking advantage of the electro-optic effect available in $LiNbO_3$ to perform the gating function. A microwave strip line and ground plane will be fabricated over the evanescent coupler

joining the feed waveguides and the resonators. Application of a voltage will change the relative propagation constant between the input/output waveguide and the resonator ring, decoupling the two. A second task of ours will be to determine the voltage-length product needed for decoupling.

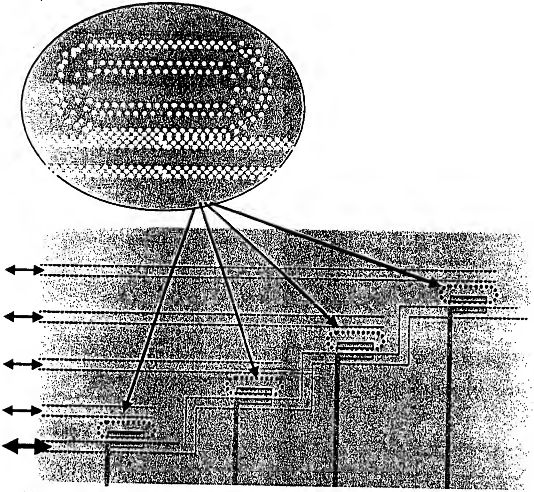

Figure 3: Schematic representation of the proposed 4-wavelength electro-optically gated optical add/drop multiplexer. The photonic crystal structures will be fabricated in epitaxial $LiNbO_3$ film. The microwave strip lines are vapor deposited and patterned gold.

If each resonator is to be responsive to only one wavelength on the ITU grid, then its free spectral range ($\nu_{FSR}=c/2nL$) need only be ~ 100 GHz. This corresponds to a maximum resonator path length of ~ 600 μm. Conventional photolithography of a much smaller length, say 60 μm, would provide sufficient center frequency accuracy. We also intend to investigate the use of the electro-optic effect to tune the resonator center frequency. The maximum resonator length to cover the C-band (1525 – 1565 nm) is about 15 μm. It should be possible to construct a defect waveguide resonator with this path length.

We estimate the device footprint for the integrated 4-channel add/drop multiplexer to be approximately 1 mm^2. If the same device were made using currently available technology (diffused waveguides in bulk $LiNbO_3$ crystals) device dimensions on the order of 10's of cm^2 would be required. The compact device is made possible only because of the excellent optical confinement possible with $LiNbO_3$ thin films, and our technology to deposit and pattern the $LiNbO_3$ photonic device structures.

1.5 Importance of the STTR Team

The investigators are uniquely suited to accomplish the goals of this program. McCaughan's group at UWM has been devoted to fabrication and characterization of $LiNbO_3$ based nonlinear optics and integrated optics for many years. This group was the first to demonstrate the use of nonlinear photonic crystals for telecommunications applications. Kuech, Saulys, and McCaughan have collaborated extensively in the areas of periodic poling mechanisms and $LiNbO_3$ film growth. Kuech is an internationally known expert both in CVD processes and reactor design. The elucidation of the film growth mechanism by Saulys and co-workers has led to the design and synthesis of more effective precursors and reactive carrier gases.

SMI is the leading US supplier of MOCVD systems for complex oxide thin films. SMI is currently developing fully integrated systems for Rotating Disc Reactor - Metal-Organic Chemical Vapor Deposition (RDR-MOCVD). We have used this technology to produce thin films and multilayers of a wide variety of complex oxide materials. Of particular interest to this effort is our related work on perovskite materials, such as $BaTiO_3$, $SrTiO_3$, $Ba_xSr_yTiO_2$ $SrBi_2Ta_2O_9$, $PbZr_xTi_{1-x}O_3$, and most notably $LiNbO_3$.

SMI and UWM have been teaming together for the past year, to develop and implement $LiNbO_3$ thin film technology. We have worked together on an MDA funded STTR effort to develop MOCVD hardware to deposit thick epitaxial $LiNbO_3$ films. We have also worked together on an Air Force funded STTR effort to develop photonic crystal devices utilizing the resulting $LiNbO_3$ films. (Please refer to Part 4.1 of this proposal for a detailed description of these efforts.) In this STTR effort, we propose to build on our previous accomplishments. We will use the process and hardware technology developed earlier to demonstrate integration of photonic devices into chip-scale optical network.

Pandanus Corporation was founded in 2001 to develop and commercialize the groundbreaking $LiNbO_3$ optical device technologies that originated at the University of Wisconsin. Pandanus original market studies indicated the potential for $500 million in component sales if fundamental $LiNbO_3$ device design limitations could be overcome. Pandanus is also building relationships with companies such as JDS Uniphase, to help with the insertion of these devices into today's fiber-optic telecommunication market. Pandanus Optical Technologies will license and commercialize the resulting photonic products developed under this effort.

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Part 2: Phase I Technical Objectives:

Phase I Objectives	
1	Demonstrate integration of photonic crystal device structures into chip scale optical networks, by building the electro-optically gated 4-channel add/drop multiplexer described herein. This work will utilize the epitaxial $LiNbO_3$ thin film technology developed earlier by the UWM/SMI team.
2	Identify critical issues for the chip scale integration technology for further development and resolution in Phase II.

Part 3: Phase I Work Plan:

Task Number	*Task Description*	*Month*
1	Produce epitaxial $LiNbO_3$ thin film samples.	1 - 4
2	Characterize the $LiNbO_3$ samples for composition and crystallographic orientation.	2 - 4
3	Fabricate the chip-scale electro-optically gated 4-channel add/drop multiplexer.	4 - 7
4	Characterize the chip-scale electro-optically gated 4-channel add/drop multiplexer.	7 - 8
5	Design additional chip scale integrated devices for further development in Phase II.	8 - 9
6	Reporting.	1 - 9

Task 1: Produce epitaxial $LiNbO_3$ thin film samples.

This task will be done at SMI. We will use the hardware developed earlier during our MDA funded STTR effort. We will also utilize the thin film deposition process technology, currently being developed under the Air Force funded SBIR effort. The UWM process initially deposits amorphous $LiNbO_3$ films. These films are then crystallized on subsequent annealing. Our standard process is to pattern the amorphous films prior to annealing, to take advantage of the higher etch rate of amorphous $LiNbO_3$.

For this task, the main challenge will be to produce epitaxial $LiNbO_3$ thin films. We have already demonstrated that homo-epitaxial $LiNbO_3$ films can be prepared on $LiNbO_3$ substrates. (Please see Part 1.3 of this proposal). For chip scale integrated optical networks, the ideal would be heteroepitaxial $LiNbO_3$ films on more practical substrates, such as sapphire or silicon. This will require the development of template layers. During this task, we will investigate template layers, deposited by MOCVD, for $LiNbO_3$ deposition on sapphire. We will anneal the as-deposited samples and characterize crystalline texture by x-ray diffraction. Our objective will be to define materials and processes to consistently produce heteroepitaxial $LiNbO_3$ thin films on sapphire substrates.

Our contingency plan will be to use a better lattice matched substrate, such as $LiTaO_3$. If we can not produce the desired heteroepitaxial films on any substrate, then we will use homoepitaxial films on $LiNbO_3$ substrates. We may still need to deposit a low index layer between the film and substrate to define the lower plane of the waveguide layer. Note that we could alternatively deposit doped $LiNbO_3$, for example with titanium, to also produce the waveguide films.

Task 2: Characterize the $LiNbO_3$ samples for composition and crystallographic orientation.

Characterization of the films will consist of x-ray diffraction to determine crystalline phase and texture, and chemical analysis to verify composition. X-ray diffraction can easily be done at either SMI or UWM. As we have found in our earlier work, accurate characterization of the Li/Nb ratio in the films is a challenge. Lithium is not an efficient emitter of x-rays. Thus techniques such as XRF and EDS do not work well. Also, SIMS is not straight forward, since lithium atoms tend to migrate in the film rather than sputter efficiently. In our current work, UWM is working to perfect ESCA techniques for analysis of $LiNbO_3$ films. If we can not identify an analytical technique that we have confidence in, the contingency plan will be to grow very thick $LiNbO_3$ films, remove and dissolve the film, and perform the analysis by atomic absorption. The characterization work will be done iteratively with Task 1. The overall objective is to produce films, with controlled composition, that consistently achieve epitaxy in the post-deposition annealed samples.

Task 3: Fabricate the chip-scale electro-optically gated 4-channel add/drop multiplexer.

This task will use the thin films developed in Task 1. The work will be done using The Wisconsin Center for Applied Microelectronics at UWM (See Part 8.1 of this proposal). The amorphous $LiNbO_3$ films will be patterned using optical photolithography and reactive ion etching. We will form the photonic crystal device structure, shown in Figure 3. The dimensions are well within the capabilities of the UWM facility. After patterning, the films will be annealed to produce crystalline epitaxial $LiNbO_3$ layers. We will then deposit and pattern gold films for the microwave strip lines, complete and package the device for testing.

Task 4: Characterize the chip-scale electro-optically gated 4-channel add/drop multiplexer.

Electro-optical characterization of the device will be done using the Integrated Optics Laboratory at UWM. (See Part 8.1 of this proposal). The objective will be to demonstrate efficient, high-speed signal multiplexing in the chip-scale integrated device.

Task 5: Design additional chip scale integrated devices for further development in Phase II.

On completion of Tasks 3 and 4, we will identify critical technical issues for chip scale integration of photonic devises using $LiNbO_3$ thin film. We will document these issues so that they can be investigated and resolved in Phase II. In addition, we will design additional chip scale networks, incorporating a variety of functional photonic crystal as well as electro-optical devices, for development in Phase II. The new networks will be designed to investigate and demonstrate resolution of the integration issues identified in Phase I.

Task 6: Reporting.

In accordance with the requirements of the Air Force, we will document all of our findings in interim reports and a final report. If invited, we will also present our plans for further development in a Phase II proposal.

Throughout this effort, both SMI and UWM will secure intellectual property rights for the hardware and processes by filing for patents, as appropriate. We will also disseminate technical information through technical publications, conference presentations, trade shows and through our product marketing efforts at SMI.

Part 4: Related Work:

4.1 UWM/SMI Research on MOCVD of $LiNbO_3$ Thin Films

UWM and SMI have recently completed an MDA funded Phase I STTR effort titled "MOCVD System for $LiNbO_3$ Thin Film Waveguide Modulators and Optical Switches". We are also finishing an Air Force funded Phase I STTR effort titled "A Scaleable Method for Fabrication Nonlinear Photonic Crystals for Ultrafast All-optical and Electro-optic Functions". The currently proposed effort will build on the accomplishments of these projects. Figure 4.1.1 shows a photograph of the MOCVD test reactor at SMI, developed for $LiNbO_3$ deposition, in part using the MDA funds.

$LiNbO_3$ thin film growth studies were carried out at SMI, using a solution of precursors in toluene, injected into a flash evaporator. The precursor mix consisted of lithium *tert*-butoxide, purchased from Aldrich, and niobium ethoxide, purified by Prof. Saulys at UW. The metals ratio was Li/Nb =1.2/1. A total metals concentration of approximately 0.25 M was employed. The depositions were carried out at a substrate temperature of 500 C and a chamber pressure of 10

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List of Suggested Reviewers or Review rs Not To Includ (optional)

SUGGESTED REVIEWERS:
Not Listed

REVIEWERS NOT TO INCLUDE:
Prof. Bruce Wessels - Northwestern University

SMI also collaborates with Prof. Wessels on MOCVD of ferroelectric thin films. SMI has confidentiality agreements with both The University of Wisconsin and with Northwestern University. We have agreed to maintain the confidentiality of the University of Wisconsin results from Northwestern University and vice-versa.

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- ☐ BEGINNING INVESTIGATOR (GPG I.A)
- ☐ DISCLOSURE OF LOBBYING ACTIVITIES (GPG II.C)
- ☒ PROPRIETARY & PRIVILEGED INFORMATION (GPG I.B, II.C.6)
- ☐ HISTORIC PLACES (GPG II.C.9)
- ☐ SMALL GRANT FOR EXPLOR. RESEARCH (SGER) (GPG II.C.11)
- ☐ VERTEBRATE ANIMALS (GPG II.C.11) IACUC App. Date ______
- ☐ HUMAN SUBJECTS (GPG II.C.11) Exemption Subsection ______ or IRB App. Date ______
- ☐ INTERNATIONAL COOPERATIVE ACTIVITIES: COUNTRY/COUNTRIES INVOLVED (GPG II.C.9) ______
- ☐ HIGH RESOLUTION GRAPHICS/OTHER GRAPHICS WHERE EXACT COLOR REPRESENTATION IS REQUIRED FOR PROPER INTERPRETATION (GPG I.E.1)

PI/PD DEPARTMENT	PI/PD POSTAL ADDRESS
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CO-PI/PD				
CO-PI/PD				
CO-PI/PD				

Electronic Signature

CERTIFICATION PAGE

Certification for Authorized Organizational Representative or Individual Applicant:

By signing and submitting this proposal, the individual applicant or the authorized official of the applicant institution is: (1) certifying that statements made herein are true and complete to the best of his/her knowledge; and (2) agreeing to accept the obligation to comply with NSF award terms and conditions if an award is made as a result of this application. Further, the applicant is hereby providing certifications regarding debarment and suspension, drug-free workplace, and lobbying activities (see below), as set forth in Grant Proposal Guide (GPG), NSF 03-2. Willful provision of false information in this application and its supporting documents or in reports required under an ensuing award is a criminal offense (U. S. Code, Title 18, Section 1001).

In addition, if the applicant institution employs more than fifty persons, the authorized official of the applicant institution is certifying that the institution has implemented a written and enforced conflict of interest policy that is consistent with the provisions of Grant Policy Manual Section 510; that to the best of his/her knowledge, all financial disclosures required by that conflict of interest policy have been made; and that all identified conflicts of interest will have been satisfactorily managed, reduced or eliminated prior to the institution's expenditure of any funds under the award, in accordance with the institution's conflict of interest policy. Conflicts which cannot be satisfactorily managed, reduced or eliminated must be disclosed to NSF.

Drug Free Work Place Certification

By electronically signing the NSF Proposal Cover Sheet, the Authorized Organizational Representative or Individual Applicant is providing the Drug Free Work Place Certification contained in Appendix A of the Grant Proposal Guide.

Debarment and Suspension Certification

(If answer "yes", please provide explanation.)

Is the organization or its principals presently debarred, suspended, proposed for debarment, declared ineligible, or voluntarily excluded from covered transactions by any Federal department or agency?

Yes ☐ No ☒

By electronically signing the NSF Proposal Cover Sheet, the Authorized Organizational Representative or Individual Applicant is providing the Debarment and Suspension Certification contained in Appendix B of the Grant Proposal Guide.

Certification Regarding Lobbying

This certification is required for an award of a Federal contract, grant, or cooperative agreement exceeding $100,000 and for an award of a Federal loan or a commitment providing for the United States to insure or guarantee a loan exceeding $150,000.

Certification for Contracts, Grants, Loans and Cooperative Agreements

The undersigned certifies, to the best of his or her knowledge and belief, that:

(1) No federal appropriated funds have been paid or will be paid, by or on behalf of the undersigned, to any person for influencing or attempting to influence an officer or employee of any agency, a Member of Congress, an officer or employee of Congress, or an employee of a Member of Congress in connection with the awarding of any federal contract, the making of any Federal grant, the making of any Federal loan, the entering into of any cooperative agreement, and the extension, continuation, renewal, amendment, or modification of any Federal contract, grant, loan, or cooperative agreement.

(2) If any funds other than Federal appropriated funds have been paid or will be paid to any person for influencing or attempting to influence an officer or employee of any agency, a Member of Congress, an officer or employee of Congress, or an employee of a Member of Congress in connection with this Federal contract, grant, loan, or cooperative agreement, the undersigned shall complete and submit Standard Form-LLL, "Disclosure of Lobbying Activities," in accordance with its instructions.

(3) The undersigned shall require that the language of this certification be included in the award documents for all subawards at all tiers including subcontracts, subgrants, and contracts under grants, loans, and cooperative agreements and that all subrecipients shall certify and disclose accordingly.

This certification is a material representation of fact upon which reliance was placed when this transaction was made or entered into. Submission of this certification is a prerequisite for making or entering into this transaction imposed by section 1352, Title 31, U.S. Code. Any person who fails to file the required certification shall be subject to a civil penalty of not less than $10,000 and not more than $100,000 for each such failure.

AUTHORIZED ORGANIZATIONAL REPRESENTATIVE		SIGNATURE	DATE
NAME: Gary S Tompa		Electronic Signature	Jan 22 2003 4:29PM
TELEPHONE NUMBER: 732-885-5909	ELECTRONIC MAIL ADDRESS: gstompa@aol.com		FAX NUMBER: 732-885-5910

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NATIONAL SCIENCE FOUNDATION
Program Solicitation/Instruction Guide Number **NSF 02-056**

SBIR PHASE I - PROPOSAL COVER PAGE

TOPIC EL	SUBTOPIC LETTER (If any) C	TOPIC TITLE **Electronics**
PROPOSAL TITLE **Low Voltage Ultrafast Traveling Wave Modulator**		
COMPANY NAME **Structured Materials Industries, Inc.**		EMPLOYER IDENTIFICATION NUMBER (EIN) OR TAXPAYER IDENTIFICATION NUMBER (TIN) **223175106**
NAME OF ANY AFFILIATED COMPANIES (Parent, Subsidiary, Predecessor)		
ADDRESS (Including addresa of Company Headquartera and zip code plus four digit extension) **Structured Materials Industries, Inc.** **201 Circle Drive** **Piscataway, NJ. 08854**		
REQUESTED AMOUNT **$100000**	PROPOSED DURATION **6**	PERIOD OF PERFORMANCE

THE SMALL BUSINESS CERTIFIES THAT:	Y/N
1. It is a small businesa as defined in the solicitation.	Y
2. It qualifies es a socially and economically disadventaged buslness es defined in the solicitation. (FOR STATISTICAL PURPOSES ONLY.)	N
3. It qualifies as a women-owned buslneas as defined in the aolicitation. (FOR STATISTICAL PURPOSES ONLY)	N
4. NSF is the only Federal agency thet hes received this proposal (or overlapping or equivalent proposal) from the small business concern. If No, you must dlaclose overlapping or equivalent proposels and awards as required by this solicitation	Y
5.SBIR: A minimum of two-thirds of the research will be performed by this firm in Phese I. STTR: It will perform et leest 40 percent of the work end the colleboreting reseerch institution will perform et least 30 percent of the work es described in the propoaal.	Y
6. The primary employment of the Principal Investigator will be with this firm et the time of the award and during the conduct of the research.	Y
7. It will permit the government to discloae the title end technical ebstact pege, plus the name, address and telephone number of a corporate officiel if the proposel does not result in an award to parties thet mey be interested in contacting the small business for further information or posalble Inveatment.	Y
8. It will comply with the provisions of the Civil Rights Act of 1964 (P.L. 88-352) and the regulations pursuant thereto.	Y
9. It has previously submitted proposals to NSF.	Y
10. It previously submitted this propoaal (which wes declined) end significant modificetions have been mede as described in the solicitation.	N
11. It has recaived Phaae II ewarda from the Federal Government. If "yes" provide e company commercialization history in the supplementary documents module.	Y

PRINCIPAL INVESTIGATOR / PROJECT DIRECTOR		
NAME **Nick M Sbrockey**		
SOCIAL SECURITY NO. **not displayed intentionally**	HIGHEST DEGREE / YEAR **PhD/1982**	E-MAIL ADDRESS **sbrockey@optonline.net**
TELEPHONE NO. **732-885-5909**	FAX NO. **732-885-5910**	WEB ADDRESS

COMPANY OFFICER (FOR BUSINESS AND FINANCIAL MATTERS)		
NAME **Gary S. Tompa**	TITLE **President**	TELEPHONE NO. **732-885-5909**

OTHER INFORMATION	
PRESIDENTS NAME **Gary S. Tompa**	YEAR FIRM FOUNDED **1994**
NUMBER OF EMPLOYEES (Including Parent, Subsidiary, Predecesaor) AVERAGE PREVIOUS 12 MO.: **12**	CURRENTLY: **12**
RESEARCH INSTITUTION NAME **University of Wisconsin-Madison**	
RESEARCH INVESTIGATOR NAME **Leon McCaughan**	
RESEARCH INVESTIGATOR TELEPHONE NO. **608-262-0311**	

PROPRIETARY NOTICE: See instructions concerning proprietary information.
Check Here ☐ If proposal contains proprietary information.

Low Voltage Ultrafast Traveling Wave Modulator

Abstract:

This Small Business Technology Transfer Phase I project will demonstrate a low-voltage waveguide modulator device, capable of operation at speeds up to 40 Gb/s. Ultimately, this device will be capable of operating at speeds up to 100 Gb/s, with drive voltages as low as 4 volts. The enabling technology for these devices is a process for deposition and patterning of single crystal $LiNbO_3$ thin films, which was invented by our STTR partners at the University of Wisconsin. Previously, the full potential of $LiNbO_3$ electro-optical devices could not be realized, due to the limitations of producing them by diffusion processes in bulk crystals. The UWM technology opens the way for a new class of electro-optical devices.

Structured Materials Industries, Inc. (SMI) has a long history of developing MOCVD systems for complex oxide film growth. UWM will work with SMI to transition the epitaxial $LiNbO_3$ film technology to commercial viability. We will also partner with a commercial supplier of electro-optical components, to provide technical guidance during the Phase I/II efforts, and for eventual commercialization of the resulting products. Together, this team is well positioned to commercialize $LiNbO_3$ thin film waveguide devices.

Statement Concerning NSF Review Criterion 2:

Fiber optic networks are being implemented in industry, defense and domestic and international telecommunications. Our proposed technology will enable new products that will add increased speed, capacity and flexibility to growing optical communications networks. We anticipate the products developed from this effort to achieve a significant market share by the year 2005. In the longer term, this technology can also be applied to devices for all-optical computing systems, which also require single crystal films of non-linear materials such as $LiNbO_3$.

Key Words:

waveguides, waveguide modulators, OEIC, fiber optics, $LiNbO_3$, thin films, epitaxy, MOCVD.

Topic Name and Subtopic:

Electronics / El-C. Photonics, Opto/Magneto-electronic Devices and Systems

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For font size and page formatting specifications, see GPG section II.C.

*Proposers may sel ct any numbering mechanism for the proposal. The entire proposal however, must be paginated. Complete both columns only if the proposal is numbered consecutively.

Low Voltage Ultrafast Traveling Wave Modulator

STTR Phase I proposal. Submitted t The National Science Foundation
In response to Topic: EL-C: Photonics, Opto/Magneto-electronic Devices and Systems
January, 2003

Part 1: Identification and Significance of the Opportunity:

1.1 Overview

To move beyond the current use of fiber optics as a point-to-point "telegraph" system will require the development of compact, high-speed, low-voltage waveguide modulators and optical switching devices. $LiNbO_3$ is a nearly ideal material for fabrication of these devices, due to its large refractive index, excellent transparency and excellent electro-optical properties. However, the full potential of this material has yet to be realized commercially. Practical $LiNbO_3$ device fabrication is thus far based on limited processing techniques applied to bulk material. The implementation of high-speed, low-voltage waveguide modulator devices has been severely limited by the absence of viable growth and patterning techniques for $LiNbO_3$ thin films.

Recently, our STTR partners at The University of Wisconsin - Madison (UWM) have developed a two-step process for producing waveguide structures in single-crystal, epitaxial $LiNbO_3$ thin films. The patented UWM process results in $LiNbO_3$ films of thickness and quality, suitable for electro-optical device applications. The UWM process also enables high deposition rates, and relatively simple sub-micron patterning, both of which impact commercial viability in a positive way. The resulting waveguide structures will serve as the basis for a new class of truly compact electro-optic devices, offering greater speeds and lower operating voltages.

In this effort, UWM and SMI propose to work together to implement and commercialize the UWM technology. UWM will perform the device design, fabrication and characterization. SMI will grow the $LiNbO_3$ thin film materials, and develop the commercial processing hardware. We will also work with potential end-users, including Pandanus Optical Technologies and JDS Uniphase, for the electro-optical device technology and ultimate device commercialization. Together, this team is well positioned to accomplish the objectives of this effort. In Phase I, we will demonstrate proof of concept for the low-voltage traveling wave modulator device based on epitaxial $LiNbO_3$ thin film. In Phase II, we will build and optimize these devices, as well as optimize the commercial hardware for their production. In subsequent activities, we will commercialize the resulting electro-optical devices and the requisite thin film processing systems.

1.2 Materials Issues for Electro-optical Devices

Modern communication is increasingly based on fiber optics. This is due to the fact that optical signals carry a higher information content (more bits/second) than conventional electrical signals[1,2]. Present day versions of electro-optical switches and modulators are based on bulk crystals of $LiNbO_3$. A third elemental species (typically titanium) is diffused into the crystal to define waveguide layers in the surface[3]. The concentration profile of these waveguide layers is

limited to the typical error-function shaped diffusion profile, and thus only graded index waveguides can be produced. As a consequence, the mode profiles of these devices are poorly optimized for electro-optic functions. In addition, the weak confinement effectively precludes densely integrated circuits (which would require serpentine structures of small radii of curvature). The end-result is devices that are large, slow, and require high operating voltages. Ideally, optical waveguide components would be fabricated from thin films of ferroelectric $LiNbO_3$. A thin film deposition technology, such as metal-organic chemical vapor deposition (MOCVD), would allow fabrication of step index waveguide structures, and permit the fabrication of structures with arbitrary dopant (e.g., Ti, Er3+, etc.) profiles. The result would be more-compact devices, with consequent lower loss, higher speeds, lower operating voltages, and a greater degree of device integration.

Previous attempts to make $LiNbO_3$ thin films have used sputtering[4,5], laser ablation[6-8], sol-gel processing[9], thermal plasma spray CVD[10,11], liquid phase epitaxy[12,13], chemical beam epitaxy[14] and MOCVD[15-22]. Although there have been many encouraging reports of epitaxial deposition, in general, the films suffer from being too thin and from having excessive optical losses. For effective waveguiding, the films thickness must be on the order of the communication wavelength (presently 1.55 microns). Epitaxial $LiNbO_3$ film deposition on sapphire has only achieved a thickness up to 2000 angstroms, due to cracking caused by the large thermal expansion mismatch with the substrate[17]. $LiTaO_3$ substrates have a much better thermal expansion match with $LiNbO_3$, but have only resulted in films up to 6000 angstroms thick[17]. Effective waveguiding also requires films with very low optical loss. Nominally, losses of less than 0.2 dB/cm are required. The sources of optical losses in $LiNbO_3$ thin films are impurities (e.g. Fe^{2+} → photorefractive effects), defects in the film, surface roughness, low oxygen stoichiometry, and crystalline inhomogeneities.[23] A chemical vapor deposition process can provide the required high purity, low-defect density films and low surface and interfacial roughness, as well as precise control over stoichiometry and doping profiles.

Another obstacle to implementing thin film $LiNbO_3$ devices is the lack of an efficient patterning technology. The high chemical stability of crystalline $LiNbO_3$ effectively precludes the use of standard photolithographic patterning techniques. As a consequence, no viable processes exist to take advantage of the large refractive index difference inherent in $LiNbO_3$/air interfaces. Demonstrated devices have thus far been based on weakly guiding waveguide structures. The UWM two-stage growth process (see below) allows high aspect ratio geometries to be produced for the first time in $LiNbO_3$. As a result, strongly confining waveguide structures (such as hairpin bends for serpentine integrated optic geometries and high-Q resonator rings) can now be fabricated in $LiNbO_3$. In addition, the index contrast ($LiNbO_3$:air = 2.2:1) is sufficiently large to permit 2D photonic bandgap devices to be constructed. The successful development of a technology for deposition and patterning of high quality $LiNbO_3$ thin films will enable a new class of truly compact electro-optic devices and circuits.

1.3 UWM Two Stage Growth Process

UWM has developed a two-stage growth process for fabricating patterned structures of crystalline $LiNbO_3$ for photonic crystal and electro-optical waveguide devices[24]. The method uses MOCVD to deposit an amorphous $LiNbO_3$ film. The amorphous $LiNbO_3$ layer can be easily patterned using conventional photolithography and standard wet or dry etching techniques. For films deposited on crystalline $LiNbO_3$, the substrate serves as an effective etch stop. For amorphous films grown on $LiNbO_3$ substrates, a post deposition anneal converts the amorphous

film to single crystal epitaxial $LiNbO_3$. We assume that similar epitaxial films would be obtained on closely lattice-matched substrates such as $LiTaO_3$.

In a recent experiment, researchers at UWM grew a 2 μm thick amorphous $LiNbO_3$ film on a z-cut $LiNbO_3$ substrate. The film was then patterned, with an orthorhombic 2D periodic pattern (7.6 x 10.1 μm^2), using standard photolithography and wet etching in dilute HF. Subsequent annealing at 1000 C resulted in the crystalline epitaxial $LiNbO_3$ structure shown in Figure 1.

Figure 1: Phase contrast Micrograph of an orthorhombic lattice (7.6 x 10.1 μm^2) patterned in epitaxial $LiNbO_3$.

Figure 2 is a cross-sectional TEM image of a similar amorphous $LiNbO_3$ film after annealing for 1 hour at 1100 C. The insert is the corresponding [0110] zone axis selected area diffraction pattern, taken from the film/substrate interface area in the image, demonstrating the single crystal epitaxial nature of the layer. The inclined lines are bend contours; the horizontal band is a thickness fringe. **To our knowledge, this is the first time a 2 micron thick epitaxial $LiNbO_3$ film has been demonstrated.** The previous largest thickness reported was 0.6 microns for an epitaxial film on $LiTaO_3$[17]. These results demonstrate the capabilities of the UWM two stage process, namely the ability to make epitaxial $LiNbO_3$ films, thick enough for practical waveguide applications, and the ability to readily pattern these films to fine geometries.

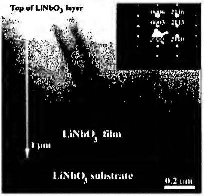


Figure 2: TEM of LiNbO3 film after annealing.

Another major impediment to the implementation of $LiNbO_3$ thin films is the consistently low growth rates observed for films deposited by MOCVD or chemical beam epitaxy (CBE). Reasonable growth rates are an important consideration for commercial viability. In the course of the thin film work at UWM, the researcher identified what they believe is the source of the low deposition rates observed for the commonly used alkoxides precursors[25]. The culprit is an autocatalytic cycle involving hydrolysis and dehydration, which generates volatile monomers of Li and Nb, instead of stable oxides of the metal. Figure 3 shows the cycle for lithium butoxide. In the absence of this cycle, the estimated growth rates for CBE would be some 5 to 10 times larger than the observed; ~ 0.2 um/h rate.

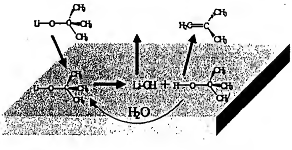


Figure 3: Model for the coupled hydrolysis and dehydration of lithium t-butoxide.

The key to overcoming this defect is to redesign the precursors such that they are either more stable in the presence of water, or decompose by a mechanism that removes the elements of water

from the surface before dehydration takes place. In principle, these next-generation precursors can be generated by either of two methodologies. A reactive carrier gas (e.g., Me_3SiCl) can be used to form more reactive precursor intermediates at the growing surface. Alternatively, and more directly, these precursor intermediates can be prepared in the laboratory and then introduced directly into the deposition chamber.

The UWM two stage process results in epitaxial $LiNbO_3$ films of useful film thickness, and the ability to pattern these films to fine geometries. We also believe we can dramatically increase the deposition rate for $LiNbO_3$, and improve the commercial viability of the process. The other potential advantages of the process are those inherent to MOCVD. We can readily control the composition and composition profile of the films. High purity $LiNbO_3$ films should be achievable. (Bulk $LiNbO_3$ crystals frequently have Fe contamination). In addition, we are not limited to growth of the congruent ([Li]/[Nb]=0.94) composition, as in the case of the bulk material. An amorphous form of stoichiometric ([Li]/[Nb]=1.00) $LiNbO_3$ should be straightforward to deposit and anneal to crystallinity. Stoichiometric $LiNbO_3$ is known to have significantly smaller susceptibility to the photorefractive effect (i.e. an increase in the material's refractive index with exposure to visible light) and a five-fold lower voltage required for poling. We should also be able to obtain engineered doping profiles (e.g. with Er^{3+}) potentially leading to a totally new class of electro-optical devices.

1.4 Device Applications

Current processing tools for $LiNbO_3$ devices are limited to thermal diffusion and electrode fabrication on bulk crystals. Our ability to grow, pattern, and re-crystallize $LiNbO_3$ thin films make it possible to produce a new generation of compact, highly integrated electro-optic devices, based on the ability to better confine both optical and microwave fields. (See Figure 4).

In this Phase I/II effort, we will focus on the development of an ultrafast, low-voltage, traveling wave modulator, as described below, since this device is anticipated to have the most significant near term market potential. However, the technology developed in this effort will be equally applicable to a wide variety of potential electro-optical devices. In the near term, these include tunable filters (also described below) and optical switches. Longer range applications include photonic crystal devices for communications and optical computing applications.

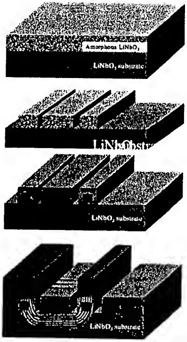


Figure 4. Schematic representing the deposition of α-$LiNbO_3$ lithography, etching, and annealing. The patterned trenches produce an improved field confinement and impedance.

<u>Low-Voltage, Ultrafast Traveling Wave Modulator</u>
Traveling wave modulators built from bulk $LiNbO_3$ crystals have been the most successful integrated electro-optic product to date. However, the inevitable demand for higher transmission bitrates (currently at 10 Gb/s), at reasonable drive voltages (under 4 volts), will require modifications to the device design beyond what can be accomplished in bulk. Our academic partner

(Leon McCaughan and UW-Madison) has carried out numerical simulations to explore thin film based devices for more near-term 40 Gb/s applications, as well as the feasibility of ultrafast modulators. As seen in the accompanying table, the patternable thin film process can be used to define trenches or wells (up to 11 μm deep) in the $LiNbO_3$ surface which improve the microwave-optical fields overlap by focusing the microwave radiation. These wells can also be used to modify the bulk dielectric properties of the $LiNbO_3$ at microwave frequencies. This latter aspect permits a better velocity matching between the two radiation fields. We note that we have recently developed the ability to fabricate very tall (in excess of 30 μm) electrode structures (via the photoresist SU-8). Calculations show that the standard gold versions of these electrodes would sufficiently reduce the microwave loss such that modulation bandwidths of up to 200 GHz can be achieved in a 4 cm long traveling wave modulator with only ~ 4 V of drive voltage. We are also exploring the use traveling wave electrodes made of copper, which is approximately 40% more conductive than gold (i.e., g_{Cu}=5.8x10^7 [Ωm]$^{-1}$ vs. g_{Au} =4.1x10^7 [Ωm]$^{-1}$). The UW lab is equipped with complete fabrication and characterization facilities, including optical and microwave test benches (with a 40 GHz network analyzer and optical spectrum analyzer). Several of the traveling wave modulator designs will be fabricated and optimized for a minimum of 47 GHz (40 Gb/s return-to-zero format).

Tunable High-Q Ring Resonators

Ridge structures provide the possibility of producing waveguides with large lateral confinement. This would permit the waveguide paths to be folded back on themselves, allowing both input and output fibers to be attached from a common end, thereby significantly reducing the device's footprint. The ability to fold waveguides would also allow long cascaded optical circuits to be folded back on themselves, reducing the device length.

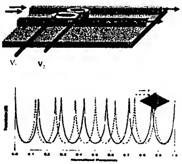
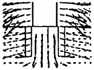


Figure 5. Electro-optically tunable filter via non-equivalent cascaded (vernier) rings.

Strong optical confinement also makes ring structures (single and cascaded) feasible, for applications such as WDM channel dropping filters, dispersion compensation, gain equalization, etc. Implementation of these structures in a nonlinear optic material such as $LiNbO_3$ would dramatically enhance the functionality of these devices: tunability by way of the electro-optic effect, frequency conversion and phase conjugation by way of 2nd order wave mixing. Figure 5 is a schematic of an electro-optically tunable filter using a pair of nonequivalent cascaded rings to provide a vernier action to the tuning[26].

Other applications of the patternable $LiNbO_3$ thin film technology can also be envisioned, including photorefractive-free thin films for visible integrated optic devices (such as RBG sources via second harmonic generation), and compact control of doping profiles during growth (e.g., for more efficient Er-doped waveguide amplifiers, vertical couplers, etc.

Table I: Model Comparisons



	Bandwidth	$V_{\pi(DC)}$	V_{3dBe}	Impedance
JDS bias-ready	15 GHz	5.5v	5.6 v	42Ω
Field-optimized	15	2.6	3.8	46
JDS	30 (40Gb/s NRZ)	6.1	*	?
Field-optimized	30	2.8	4.0	48
----------	--	--	--	--
Field-optimized	47 (40Gb/s RZ)	2.9	4.1	49

* X-cut, est. 9V, likely buffer layer for velocity match; no ridge.

1.5 Importance of the STTR Team

The investigators are uniquely suited to accomplish the goals of this program. McCaughan's group at UWM has been devoted to fabrication and characterization of $LiNbO_3$ based nonlinear optics and integrated optics. This group was the first to demonstrate the use of nonlinear photonic crystals for telecommunications applications. Kuech, Saulys, and McCaughan have collaborated extensively in the areas of periodic poling mechanisms and $LiNbO_3$ thin film growth. Kuech is an internationally known expert both in CVD processes and reactor design. The elucidation of the film growth mechanism by Saulys and co-workers has led to the design and synthesis of more effective precursors and reactive carrier gases.

SMI is the leading US supplier of MOCVD systems for complex oxide thin films. SMI is currently developing fully integrated systems for Rotating Disc Reactor - Metal-Organic Chemical Vapor Deposition (RDR-MOCVD). They have used this technology to produce thin films and multilayers of a wide variety of complex oxide materials. Of particular interest to this effort is our related work on perovskite materials, such as $BaTiO_3$, $SrTiO_3$, $Ba_xSr_yTiO_2$ $SrBi_2TaO_9$ and $PbZr_xTi_{1-x}O_3$.

Pandanus Corporation was founded in 2001 to develop and commercialize the groundbreaking $LiNbO_3$ optical device technologies that originated at the University of Wisconsin. Pandanus original market studies indicate the potential for $500 million in component sales if fundamental $LiNbO_3$ device design limitations could be overcome. Pandanus is also building relationships with companies such as JDS Uniphase, to help with the insertion of these devices into today's fiber-optic telecommunication market. Pandanus Optical Technologies will license and commercialize the resulting photonic products developed under this effort.

Part 2: Phase I Technical Objectives:

The mission for the UWM / SMI team is to bring the UWM process technology to market. We will accomplish this in two steps. In Phase I, we will demonstrate technical feasibility. In Phase II, we will develop actual products. We will also partner with commercial organizations engaged in the development of fiber optic communications products. Pandanus Optical Technologies, of Madison, WI will provide technical input to the initial stages of this effort, and license and commercialize the resulting electro-optical devices during later program stages.

The UWM / SMI / Pandanus team is uniquely qualified to accomplish the goals of this program. Each brings specific capabilities to the effort. UWM provides the thin film process technology, and materials and device characterizations capabilities. SMI brings expertise in development and commercialization of MOCVD film deposition systems. Pandanus provides knowledge of optical communications technology and products. In addition, we will also partner with JDS Uniphase, who brings worldwide distribution capabilities to this effort.

The following Table summarizes the team's objectives for the Phase I program, as well as for Phase II and the eventual commercialization.

Phase I Objectives	
1	Optimize the UWM two-step process using one of SMI's scaleable test MOCVD reactors, and produce $LiNbO_3$ thin films for subsequent characterization at UWM. The Phase I targets for the film deposition work are epitaxial $LiNbO_3$ films, at least 2 microns thick, deposited at rates of at least 1 micron/hour.
2	Demonstrate properties of the $LiNbO_3$ films that meet the requirements for compact, high-speed electro-optical devices. The Phase I targets are patterning and waveguide formation in the epitaxial $LiNbO_3$ films, demonstration of optical losses of 0.2 dB/cm or less, and fabrication and of the 40Gb/s low voltage (4 V or less) traveling wave modulator.
Phase II Objectives	
1	Develop and refine the $LiNbO_3$ MOCVD deposition process, to produce films suitable for electro-optic device fabrication, on wafer sizes up to six inches. The Phase II targets are epitaxial $LiNbO_3$ films, greater than 5 microns thick, deposited at rates of 5 micron/hour or better, with thickness uniformity better than 5% and optical losses less than 0.2 dB/cm.,
2	Develop and demonstrate $LiNbO_3$ thin film low-voltage ultrafast waveguide modulator devices. The Phase II targets are data rates up to 200 Gb/s, at drive voltages of less than 4 volts.
Commercialization Objectives	
1	Commercialize $LiNbO_3$ thin film electro-optical devices through strategic partnerships with companies such as Pandanus Optical Technologies and JDS Uniphase.
2	Commercialize the MOCVD hardware to implement the UWM two step deposition process for fabrication of thin film $LiNbO_3$ devices.

Part 3: Phase I Research Plan:

Task Number	*Task Description*	*Month*
1	Refine and demonstrate amorphous $LiNbO_3$ deposition using a scalable test MOCVD reactor at SMI.	2 - 4
2	Refine and demonstrate the annealing technology to produce epitaxial $LiNbO_3$ films from the amorphous deposits.	3 - 5
3	Fabricate waveguide structures in the resulting epitaxial LiNbO3 films. Characterize the films chemical, physical and optical properties and demonstrate their suitability for the low-voltage ultrafast waveguide modulator.	4 - 5
4	Deliverable - Phase I final report and Phase II proposal.	6

<u>Task 1:</u> Refine and demonstrate amorphous $LiNbO_3$ deposition using a scalable test MOCVD reactor at SMI.

SMI will perform process optimization, starting with the UWM process and precursors. We will use low cost substrates (such as silicon or sapphire) for the initial process development. We will then use either $LiNbO_3$ or $LiTaO_3$ single crystal substrate, for demonstration of the optimized epitaxial film properties. The primary intent of this task will be to demonstrate a process to deposit amorphous $LiNbO_3$ films, at least two microns thick, at growth rates of at least one micron/hour. In the course of this activity, we will identify any hardware issues related to the scale-up of the process to commercial wafer sizes and production volumes. We will show proof of concept that we can resolve all scale-up issues. We will also investigate different $LiNbO_3$ film compositions, including both the congruent ([Li]/[Nb] = 0.94) and stoichiometric ([Li]/[Nb] = 1.00) film compositions. Bulk $LiNbO_3$ materials are limited to the congruent composition. However, the MOCVD process should allow us to deposit films of any composition. Selected films from this task will be provided to UWM for evaluation as described in Tasks 4 and 5 below.

<u>Task 2:</u> Refine and demonstrate the annealing technology to produce epitaxial $LiNbO_3$ films from the amorphous deposits.

UWM will refine and demonstrate the annealing technology to produce epitaxial $LiNbO_3$ films from the amorphous deposits produced at SMI in Task 1. Because $LiNbO_3$ is subject to Li_2O out-diffusion during high temperature anneals. They have recently designed an annealing station which contains an overpressure of Li_2O. The goal of this task will be to demonstrate epitaxial films, of 2 micron thickness or greater, without stress related failures such as cracking. We will also demonstrate a process to pattern the amorphous deposits, and anneal them to produce pre-defined structures in the epitaxial $LiNbO_3$ films.

The resulting thin films will be characterized as needed at UWM. Chemical characterization will be made using SIMS. Structural characterization will be made principally via X-ray diffraction, with a limited number of TEMs to determine defect density. Maker Fringe analysis (a form of surface second harmonic generation) will be used to determine and monitor the Li/Nb ratio. These characterization results will be provided as feed-back to the on-going process development work at SMI.

Optical characterization will consist of fabricating channel waveguides and making propagation loss measurements, and determining the magnitude of the nonlinear optic coefficients via the electro-optic effect (e.g. using a standard Mach Zehnder waveguide interferometer). Our targets for this task are to obtain films with optical loss of 0.2 dB/cm or less, and electro-optic properties approaching that of bulk $LiNbO_3$. UWM is fully equipped to carry out all aspects of this characterization. See the Facilities Section of this proposal for a description of laboratory capabilities at UWM.

Task 3: Fabricate waveguide structures in the resulting epitaxial LiNbO3 films. Characterize the films chemical, physical and optical properties and demonstrate their suitability for the low-voltage ultrafast waveguide modulator.

Selected samples of the most promising epitaxial $LiNbO_3$ films will be fabricated into simple waveguide structures and devices at UWM. We will also provide samples to our partners at Pandanus Optical Technologies and JDS Uniphase (and other potential customers/partners that we identify) for their evaluation. The deliverable for this task will be verification that the films have the necessary properties for application in electro-optical devices, and a plan for the development of these devices in the Phase II effort.

Task 4: Reporting.

In accordance with the requirements of NSF, we will document all of our findings in a final report. If feasibility is demonstrated, we will also present our plans for further development in a Phase II proposal.

Throughout this effort, both SMI and UWM will secure intellectual property rights for the hardware and processes by filing for patents, as appropriate. We will also disseminate technical information through technical publications, conference presentations, trade shows and through our product marketing efforts at SMI.

Part 4: Commercialization Potential:

The product. The focus of this effort will be low voltage ultrafast traveling wave modulators. However, the successful implementation of the UWM two-step process will also enable many other electro-optical devices, meeting both near term (40 Gb/s), as well as future (100 Gb/s and beyond) high speed communications demands. This technology could also enable eventual optical computing and all-optical communications products. The proposed technology will enable not only these devices, but a robust and production-worthy technology for their manufacture.

At the conclusion of the Phase II effort, we will commercialize the low voltage ultrafast modulator devices, and evaluate the other opportunities as appropriate. In addition to the devices, we also plan to sell high-volume manufacturing equipment for the epitaxial $LiNbO_3$ thin film processing, and non-exclusive licenses for the process technology.

The Markets. The world wide market for telecommunications components and equipment is anticipated to expand, after an initial decrease in 2001 and 2002. An approximate 35% annual growth rate is predicted over the next four years[27]. The market estimates reach $23 billion by 2005. In addition to the components market, there is also a market for the infrastructure (such as manufacturing equipment) to support this market. We plan to capitalize on these trends.

The Competition. The main competition to the proposed technology is from present day devices for optical telecommunications. These are primarily devices based on bulk $LiNbO_3$ crystals. The technology proposed in this effort will result in devices that are smaller, cheaper, operate at lower voltage and are capable of higher speeds. However, these technical advantages alone will not guarantee success, unless there is also a market pull for these products. Such market pull is difficult to see right now, due to the present overcapacity in the US telecommunications industry. However, the need for high speed optical communication devices still exists; particularly for specialized applications such as for military and homeland defense. Also, the demand for telecommunications products is still increasing. This market increase is real, although it is not readily apparent due to the present industry overcapacity. None-the-less, market demand will eventually catch up, most likely in the next 1 to 2 years. When the market does catch up, those companies with a technology advantage will be in a very good position.

MOCVD is the only thin film deposition technology that can potentially meet the needs of these products. SMI is the leading company specializing in MOCVD of complex oxide thin films. Our main competitors are Emcore, www.emcore.com, and Aixtron, www.aixtron.com, both of whom specialize in MOCVD systems for compound semiconductors. While both Emcore and Aixtron have competitive hardware technology, neither has significant process technology for thin film oxides. In addition, neither has significant expertise in end-uses involving piezoelectric or ferroelectric thin film materials.

The Commercialization and Financing Plan. A key element of our commercialization strategy is to first start with the right team. The team for this effort includes The University of Wisconsin at Madison, which has one of the foremost research efforts in the US for electro-optical thin films for device applications. UWM brings considerable expertise and existing process technology to this effort. Our team also includes Structured Materials Industries, Inc., who is the leading US company specializing in MOCVD systems for complex oxide thin film deposition. SMI brings expertise in the deposition hardware to this effort. Completing the team are Pandanus Optical Technologies and JDS Uniphase, who bring expertise in electro-optical devices and an existing market presence.

Our strategy will be to prove technical feasibility in Phase I, then develop the products in Phase II. In subsequent work, we will commercialize this technology at no cost to the government. We will first protect any intellectual property, by filling for the appropriate patents. Then, SMI will place a beta-system MOCVD reactor at Pandanus. Pandanus will use the SMI system and the UWM process technology to produces prototype traveling wave modulator products, and begin sampling these to customers. We will learn as much as we can from this development work and publicize the results as much as permissible. SMI and Pandanus will work collaboratively to disseminate the results of our efforts through (1) technical and trade publications, (2) advertising and presentations at trade shows, (3) presentations to potential customers and collaborators, (4) site visits to promising customers, (5) marketing through representatives and our respective websites and (6) through press releases. We will use this publicity to identify potential customers and other potential products that can be developed using the epitaxial $LiNbO_3$ thin film deposition and patterning technology.

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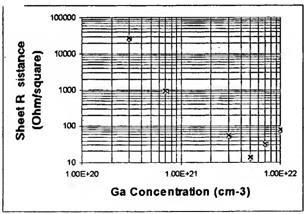
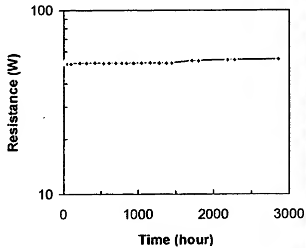
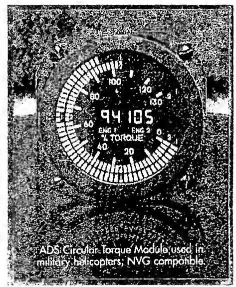
Focused Application TCOs

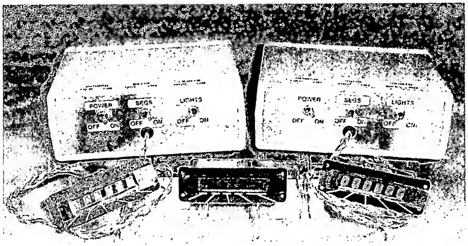
Displays require Transparent Conductive Oxides (TCOs) for contact layers; heaters and insulators. (Oxides also have a strong potential as efficient phosphors)

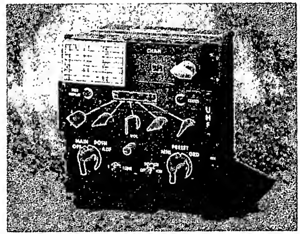






Transparent Conducting Display Oxides

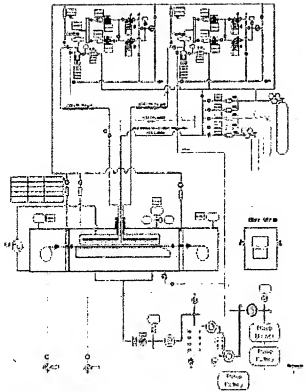




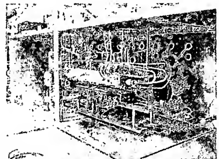
Advanced military and commercial displays require higher durability optical coatings

MOCVD Tape Coating System

Superconductors – Batteries – Circuit board components – an so on

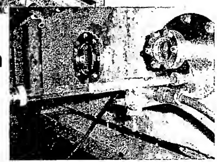


MOCVD System Schematic



MOCVD Assembly

UV Option

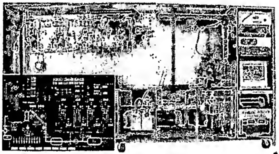


Plasma Option

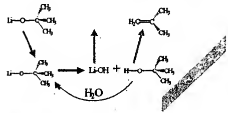
$LiNbO_3$ *Photonic Crystal Devices*

Structured Materials Industries, Inc.

University of Wisconsin
& Corporate Sponsor

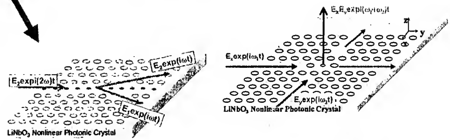


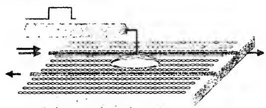
- Oxide MOCVD systems
- Wafer scaling



- Surface Chemistry
- PC design & fab.
- Device modeling

Phase contrast micrograph of an orthorhombic lattice (7.6 x 10.1 μm²) patterned in $LiNbO_3$





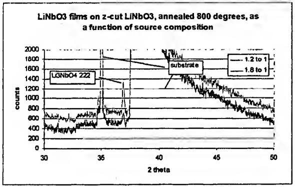
Gated Wavelength Filter

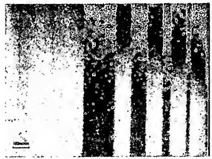
Targeted Customers

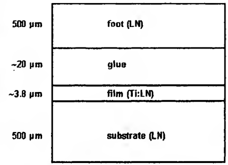
JDS, Spectallis, ...

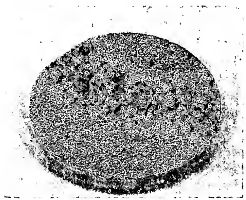
- Quantum communication
- Quantum computation
- Optical Data Manipulation
- Optical Logic

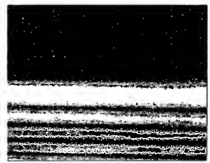
LiNbO$_3$ properties





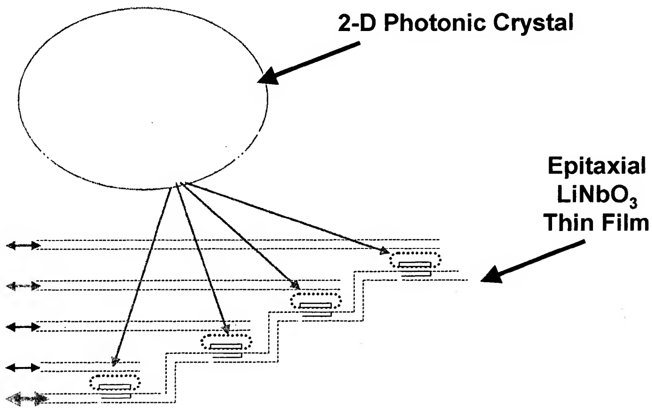






Light guiding in Ti-doped LiNbO$_3$

Electro-Optically Gated Add/Drop Multiplexer



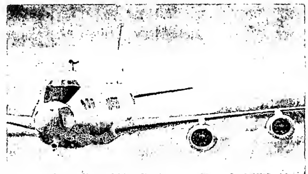
Very Low Absorption Coatings for High Power Laser Optics

Laser Damage Resistant Coatings for the Airborne Laser (ABL)

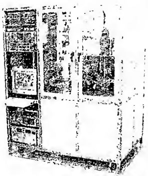
Requirements

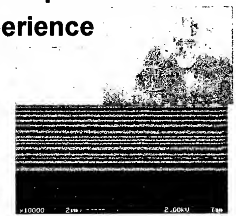
- Very Low Optical Absorption
- Low Residual Stress
- Refractory Material, Temperature and thermal Shock Resistant
- Phase I concepts proven, Phase II scale process

Missile Defense System



SMI Reactor Capabilities & Coating experience





RPE-MOCVD Coatings Properties

- High Purity
- Excellent Stoichiometry (Low Optical Absorption)
- Low Residual Stress
- Wide Range of Materials